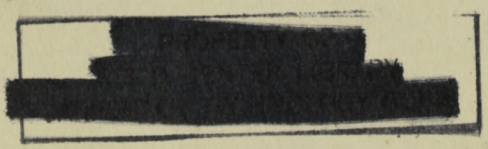
RESEARCH & METHODS of ANALYSIS of IRON AND STEEL

[redacted]



COPYRIGHT
AMERICAN ROLLING MILL CO.
MIDDLETOWN, OHIO
1920

PRESS OF
THE GIBSON & PERIN CO.
CINCINNATI, OHIO

669.9
Am 35

Research and Methods of Analysis

of

IRON AND STEEL

at

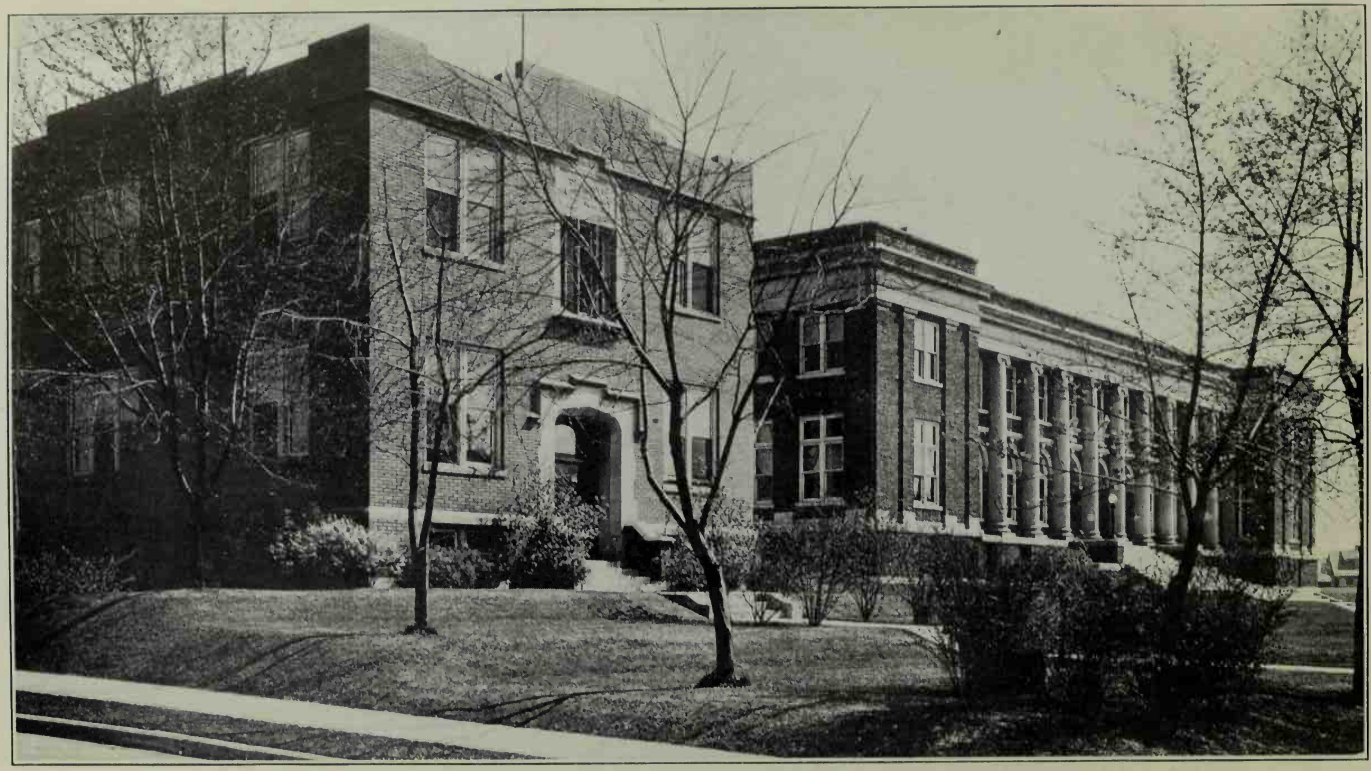
ARMCO

Second Edition
Price $4.00

DISCARDED
R&D LIBRARY

THE AMERICAN ROLLING MILL COMPANY
MIDDLETOWN, OHIO
1920

527



Research Laboratory and General Office of the American Rolling Mill Co., Middletown, Ohio

669.93
Am 35

Preface to Second Edition

THE first edition of this book appeared in 1912. The supply was soon exhausted, and continued demands have made it seem desirable to issue a second edition.

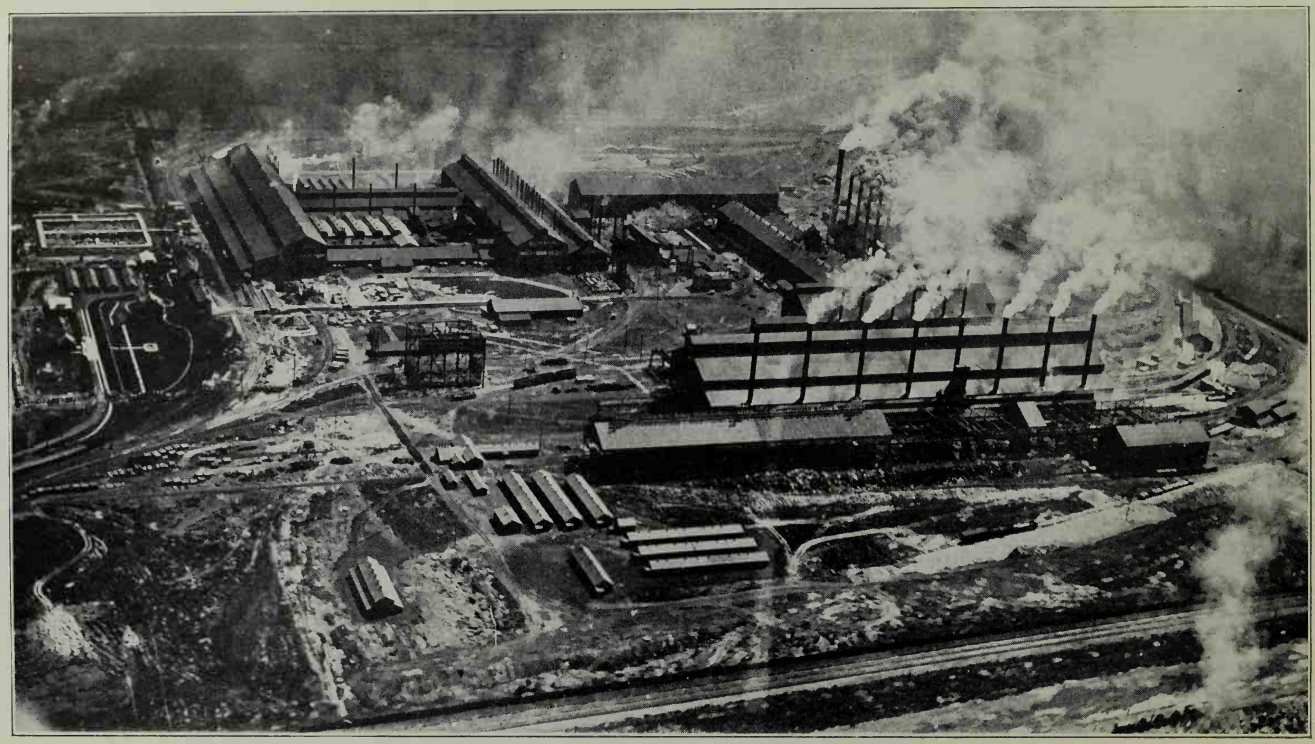
The methods described are particularly adapted to the analysis of "Armco" products.

Where well-known methods have been described, we have omitted details which are well understood by the skilled chemist. Where new methods are described, we have entered into minute details.

The second edition has been entirely rewritten, many methods have been added, and the entire scope amplified by the addition of new material.

ARMCO RESEARCH
MIDDLETOWN, OHIO
1920

527



Aeroplane View of "Armco" East Side Works, Middletown, Ohio. The American Rolling Mill Company has four plants, two of which are located at Middletown, approximately one mile apart.

INTRODUCTION

EVER since its inception, The American Rolling Mill Company has been a leader in the adaptation of science to practical problems of steel making.

The company was organized to manufacture special grades of sheet metal suited to the needs of exacting users.

A well-equipped research laboratory was, therefore, essential to the carrying out of its manufacturing program.

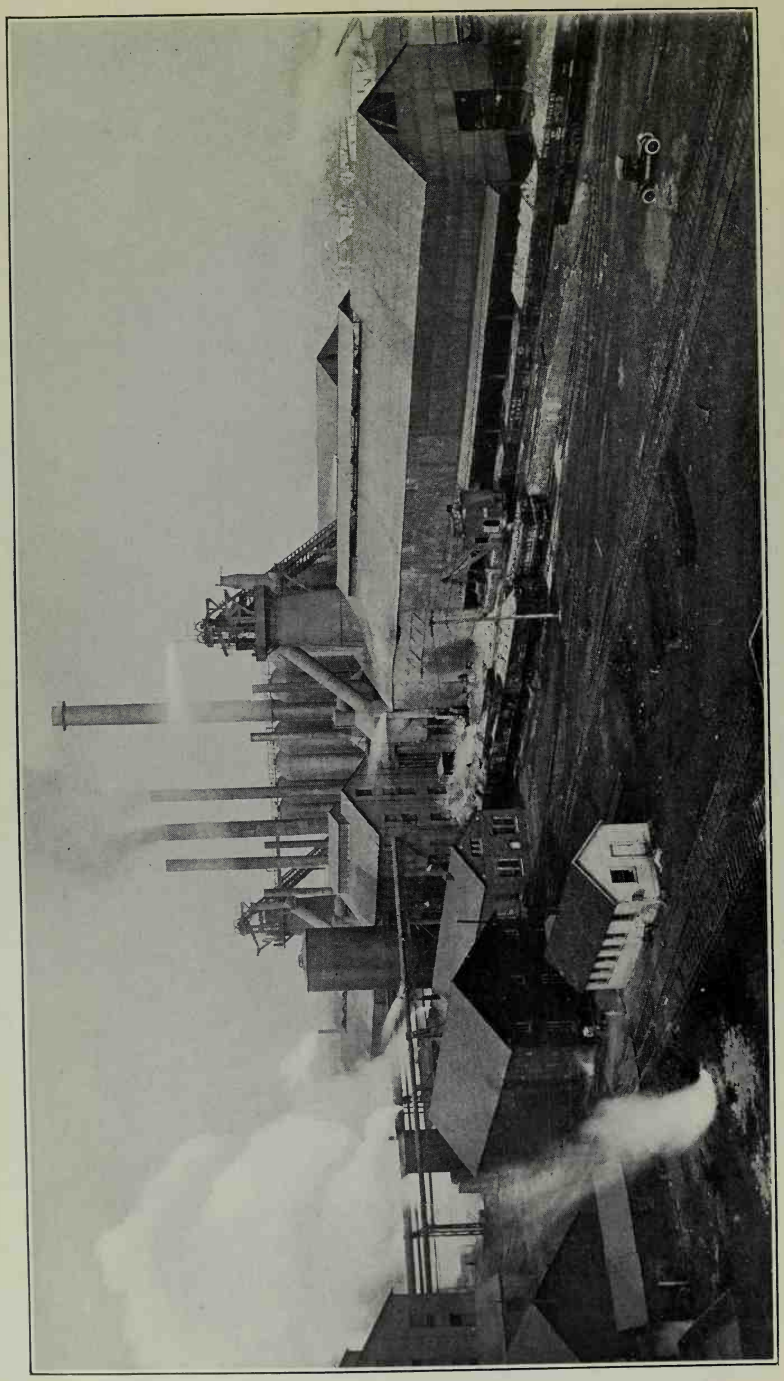
In the establishment of such a laboratory, a new era was begun covering the manufacture of high grade iron and steel sheets and other metal products.

In 1903 the development of various grades of electrical sheets for transformers, motors, generators, etc., was begun. Most satisfactory results were secured and research work on this most important line of manufacture has been continued up to the present date.

In 1908 for the first time in metallurgical history and contrary to established theories as laid down in authoritative metallurgical text books of the day, a commercially pure iron made in a modern open hearth furnace was produced.

This new metal was soon found to be superior to Bessemer and Open Hearth Steel, in the essential properties of Purity, Rust-Resistance, Welding, Conductivity, and Enameling. Its metallurgical and practical development is unquestionably largely responsible for the improvement made to date in the rust-resisting qualities of the various grades of iron and steel sheets now being manufactured.

In the early days, Chemistry had not in a majority of cases been applied to practical steel making beyond the determination of the elements known as "The Big Five" (Sulphur, Phosphorus, Carbon, Manganese, and Silicon). Today through research development, it is known that



Armco Blast Furnaces at Columbus, Ohio where the Pig Iron is Manufactured

gases such as Nitrogen and Hydrogen are elements to be considered in rust-resistance and other qualities of iron and steel.

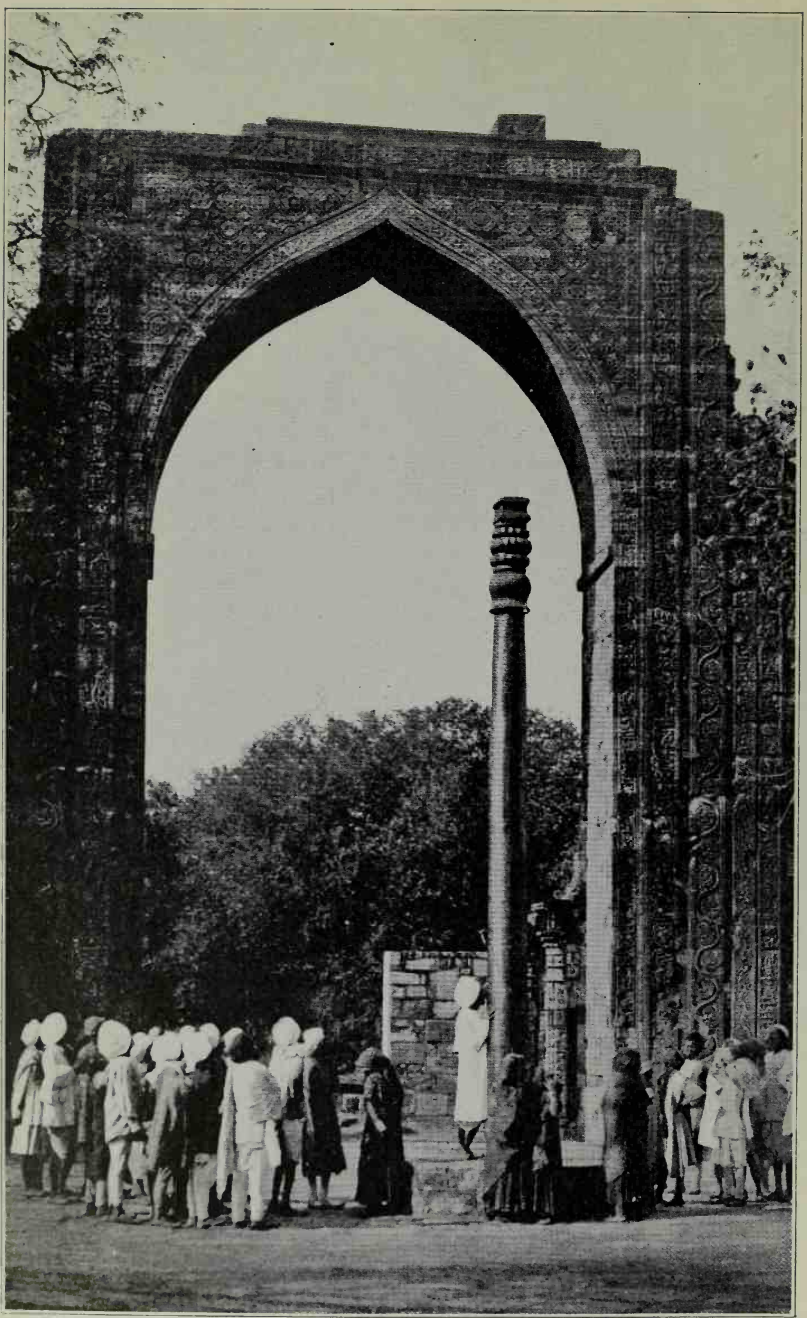
A close study of protective coatings is another branch of metallurgical research. Work along this line has been done to show the effect of impurities in the coating. It is a recognized fact that pure galvanized coatings are much more resistant to the elements of corrosion than are impure coatings, and experiments are being constantly carried on to further improve the quality and character of these coatings.

Degasification of metal was not thought of a few years ago and yet today it is considered of very great importance. A modern research laboratory can now determine the gases in the metal, and with this information, it is possible to maintain control of the gas content in the manufacture of the product. The Research Department of The American Rolling Mill Company was the first to make practicable, various methods for gas determination in Iron and Steel.

New uses for pure iron and other special sheet metal products require a constant expansion of research work, covering such lines as vitreous enameling, japanning, welding, heat treatment, forging, and casting, all of which offer wonderful fields of usefulness for the chemist and metallurgist.

During the last ten years many grades of high polished sheets have been produced for the use of the automobile, furniture and other products. These sheets must not only have a very high finished surface, free from defects of every kind, but they must stand all sorts of drawing and spinning operations unknown to the maker and user of sheet metal just a few years ago.

The modern research laboratory has been largely responsible for all of these developments and it needs no prophet to foresee that many new alloys and other products, the result of special manufacture and treatment, will be developed from time to time to meet the exacting demands of industrial progress.

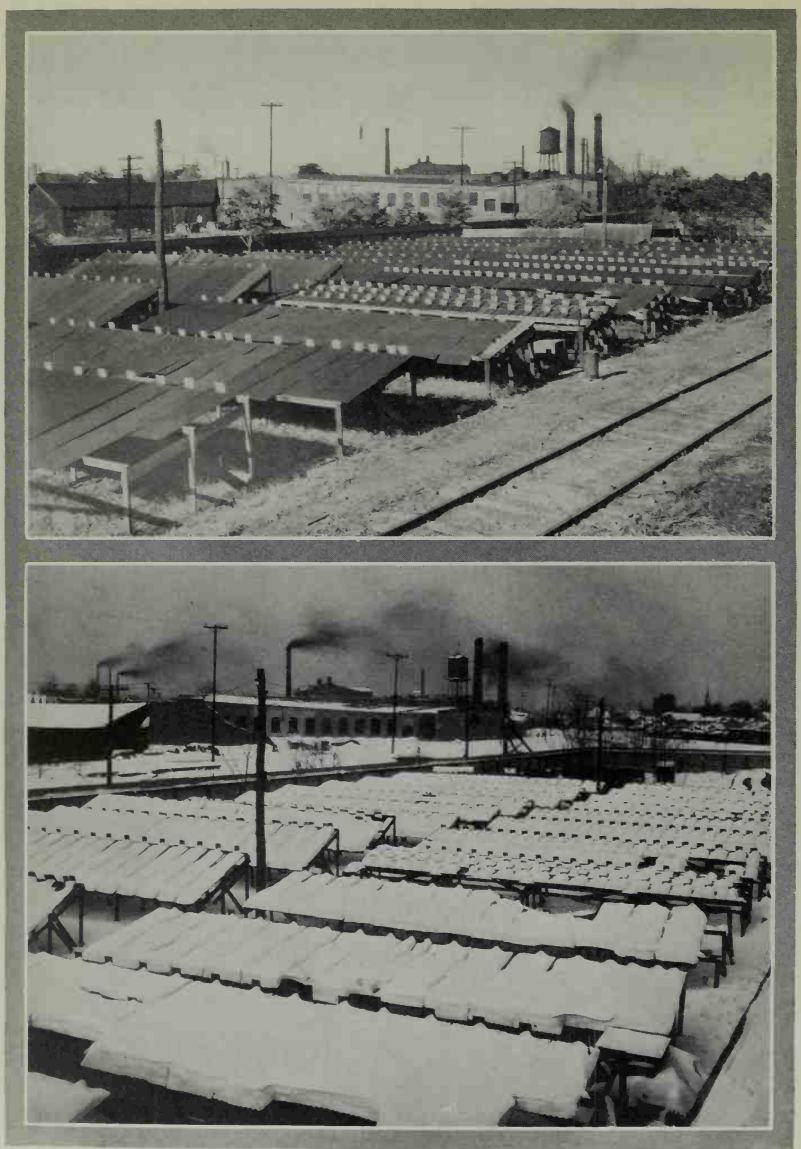


The Iron Pillar at Delhi, India

IRON PILLAR OF DELHI, INDIA, 1600 YEARS OLD

Described by Sir Robert Hadfield In the 1912 Journal of the Iron and Steel Institute. He shows it to be pure iron of the following analysis;

Silicon	.046
Sulphur	.006
Phosphorus	.114
Carbon	.080
Manganese	nil
Copper, Etc.	.034
Iron	99.720



In the study of corrosion problems it has been necessary to provide extensive proving grounds. This view shows one of the proving grounds where various metals are exposed to atmospheric conditions

ANCIENT IRONS AND MODERN RESEARCH

IN the British Museum and elsewhere are many interesting specimens of age old irons that have resisted the "rust of time". Some of them have been taken from the tombs in the pyramids of Egypt. Their existence spans a period of 4000 years to the modern world in which steel and iron plays so large a part.

For years, scientists have been seeking the secret of rust-resistance of iron and steel. They have analyzed such ancient irons as came to light for a complete understanding of their contents, they have studied their grain structure with the microscope, they have determined the gas content, and have taken into consideration the primitive methods of manufacture as compared with those of today. Out of all this has come the deductions of modern science, that is marking the pathway of progress.

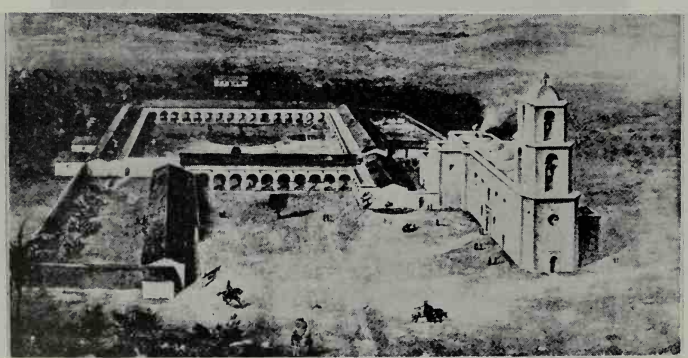
The American Rolling Mill Company has been an earnest investigator of the causes of corrosion. In the Research Department at Middletown is a museum of old and interesting nails and odd bits of old iron. The history together with the physical and microscopical analysis of each is carefully investigated and recorded in the archives, while the specimens are laid away under glass cases to awe the visitor with their antiquity. In fact, no sooner does an interesting example of the old iron come to light, than someone will suggest sending it to the Research Department of The American Rolling Mill Company at Middletown, Ohio, for analysis. The study and analysis of these old irons has brought world wide recognition of Armco research work.

Among the interesting old iron curios that have been sent to Middletown for analysis is a piece of iron cut from the "Merrimac," after having been in the water for more than one-half a century. It is historically interesting because of the famous "Monitor and Merrimac" fight in Hampton Roads during the Civil War.

HAND FORGED NAIL 145 YEARS OLD
MADE BY INDIANS AND USED
IN MISSION

ANALYSIS

Sulphur	.005
Phosphorus	.057
Carbon	.015
Manganese	.015
Copper	trace
Silicon	.048
Oxygen	.109
Nitrogen	.006



MISSION SAN JUAN CAPISTRANO, CALIFORNIA.
BEFORE THE EARTHQUAKE OF 1812
THE FIRST FOUNDING OF THIS
MISSION TOOK PLACE
Oct. 30, 1775.

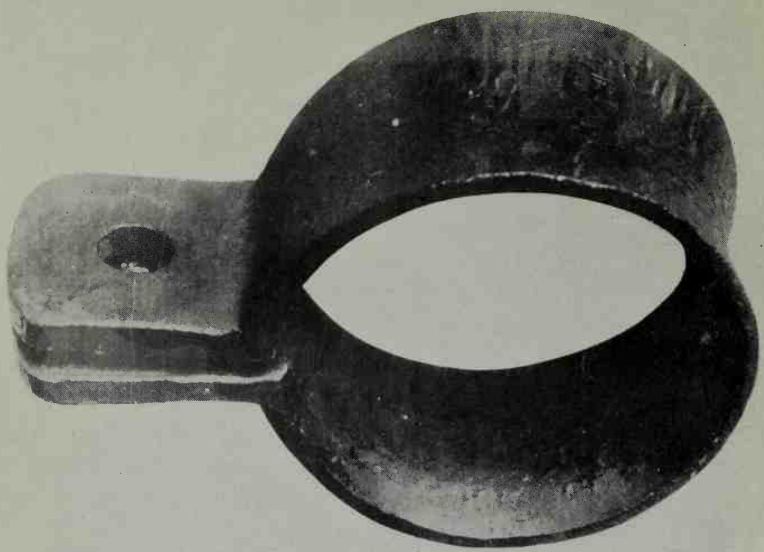
Along side this is a collection of nails from the Wayside Inn at Sudbury, Mass. Nails from the famous Fairbanks homestead at Dedham, Mass., share their interest with nails from historic old missions of California. Coffin nails buried 100 years ago and still in a state of perfect preservation have their own story to tell.

In the corner of the room is still another notable example of rust-resisting iron—the old iron links taken from the Newburyport bridge at Newburyport, Mass. Notwithstanding the fog and dampness of the New England seacoast, when the bridge was taken down in 1910, after 100 years service, the heavy iron links were apparently as good as the day they were installed.

The collection of interesting specimens is being added to every day. Recently an old iron nail was sent to the Research Department of The American Rolling Mill Company, which was picked up out of the shell-torn ruins of the home of John Calvin, the great Reformist, at Noyon, France. The house was known to be at least 400 years old and yet the huge iron nail used in its construction showed no sign of corrosion.

The Research department at Armco also prizes most highly specimens in its possession which were taken from the Pillar of Delhi, India—the most notable example of old iron in the world today. This wonderful relic of worship dates back sixteen hundred years, and still stands today defying the elements and the "kisses" of worshipers, with no sign of disintegration by rust or corrosion.

Over these specimens of old iron, the scientist works like the etymologist over his insects, searching for hidden secrets.



IRON BAND TAKEN FROM A CANNON CAPTURED FROM THE BRITISH IN THE BATTLE OF TICONDAROGA. 1775-1777

TEST NO. 1036 FILE 51

ANALYSIS

Sulphur	.005
Phosphorus	.069
Carbon	.010
Manganese	.010
Copper	.080
Silicon	trace
Oxygen	.063

RESEARCH ON CORROSION

DURING the past few years a number of papers have been published by various investigators both in America and Europe, presenting the results of corrosion tests made under varying conditions of exposure, both in the laboratory and in the field. The size of the test pieces in the hands of the separate investigators have ranged from small specimens which could be weighed on a chemical balance up to full size commercial sheets exposed to the natural wet and dry conditions of the outdoor atmosphere.

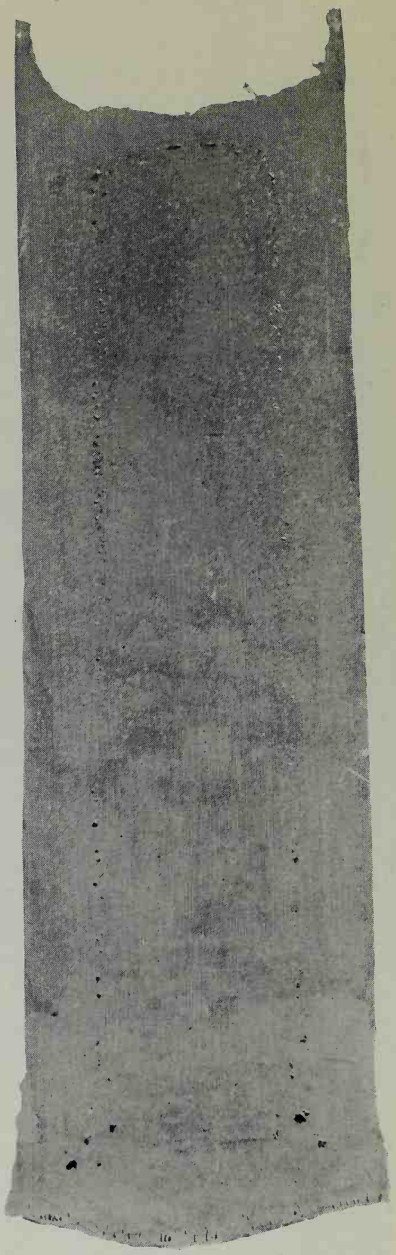
Total and partial immersion tests in various media, with loss of weight during progressive corrosion, have been carefully recorded and plotted in the form of curves, and on these data in some cases very sweeping conclusions have been drawn. Nearly all the investigators have interested themselves in the possible beneficial or deleterious effect of more or less minute quantities of some special impurity or element, or in some grouping or variable combination of the usual constituents of commercial metals.

Thus far all the experimenters have contended themselves with a discussion of the possible effect of solid constituents, without taking into account the possible effect of impurities of a gaseous nature, which are always more or less associated with iron and steel, such as carbon monoxide, carbon dioxide, oxygen, hydrogen, and nitrogen.

It is self evident that the corrosion of iron and steel depends very largely upon the amount and character of surface exposed. It is obvious that metal rolled from spongy and porous steel, some of the blowholes in which extend to the surface, will be more susceptible to the action of water and oxygen, and also the nature of such rust will be different from that produced on sound metal. As unsound metal corrodes, a loose rust is produced on account of the tendency of the occluded gases to escape. A dense, sound metal, on the other hand, will form a dense, closely adherent rust, and the rate of progressive corrosion will be very materially reduced.

The fact is not generally enough understood, that gases such as nitrogen, hydrogen, oxygen, and carbon monoxide may be associated with iron and steel in three separate and distinct ways. Gas may be present as an atmosphere enclosed in open blowholes, pipes or seams,

Section through center of Steel Ingot, showing subcutaneous and deep seated blowholes.



Section through center of "Armco" Ingot Iron, ingot, showing comparative freedom from blowholes.

which may be large enough to see with the naked eye or so small as to require a microscope to resolve them. Again, free gas may be occluded among the grains or molecules of the iron, and be present in quantity even though no microscope is capable of discovering its presence. Finally, gases may be present in chemical combination with iron or manganese, or some other constituent, to form either dissolved or segregated nitrides, hydrides, oxides, or carbonyl compounds.

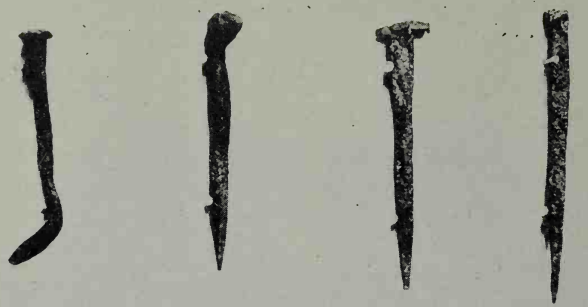
That all three forms of gas inclusions affect the resistance of all metals to corrosion, there are strong ápriori reasons for believing, even if there was no evidence as to fact in support of such a view.

We have sawed ingots of iron and steel in two longitudinally, so that the soundness could be observed. The photographic cross sections of these experimental ingots are given.

The atmospheric exposure tests which we are conducting, containing more than six hundred full size corrugated, 26-gauge sheets, first began to exhibit failures after about 13 months, exposure. The Bessemer Steel sheets have all failed in periods ranging from 13 to 34 months.

We have now to consider why it is that Bessemer steels as a general rule rust more quickly than open hearth steels. As far as we are aware, no explanation of this frequently observed phenomenon has heretofore been advanced. Bessemer steel is made in America exclusively in acid lined converters, and, as this lining has no dephosphorizing action, American Bessemer Steels usually run a little higher in phosphorus than the general run of basic open hearth steels. This is not, however, always true, and an analysis of a steel for its phosphorus content is by no means a definite proof of its method of manufacture. However, supposing that the general run of Bessemer steels made in America run somewhat higher in phosphorus than the general run of open hearth, is there any evidence that slightly higher phosphorus would account for any difference in corrosion resistance of the two types? As a matter of fact, phosphorus is one element of impurity in steel that has not at some time or another been held to be a prime factor in rapid corrosion.

It seems to be very well assured that the tendency towards rapid corrosion, noted in the case of most Bessemer steels, must be due to some element of difference beside the usual slightly higher phosphorus content.



IRON NAILS ABOUT 281 YEARS OLD TAKEN FROM FAIRBANK'S HOUSE. DEDHAM, MASS.

TEST NO. 4541 FILE 103

ANALYSIS

Sulphur	.009
Phosphorus	.005
Carbon	.015
Manganese	.020
Copper	.016
Silicon	.029
Oxygen	.060
Nitrogen	.005
Iron	99.841

Going back to the differences in method of manufacture of Bessemer and open hearth metal, it at once occurs to us that the former type has blown through it, while it is molten and in the process of conversion, enormous quantities of air, more or less laden with moisture and gases. Of the two types of metal, therefore, Bessemer should be more prone to run a high gas content in the cooled and finished product than properly made open hearth steel. If this is true, the questions that at once arise are, can gas content be a factor in the relative corrosion resistance of Bessemer and open hearth metals, and further than this, is the self same factor an important one in considering relative corrosion resistance of different types of basic open hearth metal.

Fortunately, there is some recent scientific data of a most interesting nature, which shows in a most conclusive manner what an enormous quantity of gases associate themselves with some types of commercial steel and modify or qualify their physical characteristics.

In 1914, L. Baraduc Miller published in full in the Carnegie Scholarship Memoirs of the British Iron & Steel Institute a report of progress on the study of gases occluded in liquid steel. The steel which was made by the basic Bessemer process was cast into 1000 lb. ingots and allowed to cool in a vacuum, while other ingots from the same heat were cooled under atmospheric pressure in the usual manner generally practiced in metallurgical operations.

One ingot of the vacuum treated steel yields 1159.8 liters of gas measured at atmospheric temperature and pressure. This gas on analysis was found to have the chemical and volume composition as shown in the following table:

	Per Gross Volume	Per Cent	Per Ton of Steel
	Litres		Litres
Carbon Dioxide	42.2	3.6	76.7
Oxygen	10.6	0.9	19.3
Carbon Monoxide	352.2	30.5	640.3
Hydrogen	604.3	52.2	1098.7
Methane	2.4	0.2	4.3
Nitrogen	147.7	12.7	268.5

This table shows the astonishing fact that more than 2100 liters of gas, or about 75 cubic feet per ton of steel was liberated from the vacuum treated metal. It is also shown that the principal gases, hydrogen, carbon monoxide and nitrogen, play an important role in

MICROSTRUCTURE OF LONGITUDINAL SECTION

IRON CHISEL OF ANCIENT ORIGIN ABOUT 1400 YEARS OLD

Described by Sir Robert Hadfield in the 1912 "Journal of The Iron and Steel Institute."

the metallurgy of steel. In another table Baraduc Miller gives the following data to show the physical character of the vacuum treated steel compared with metal from the same ladle poured and cooled in the usual manner:

	Test piece of metal not treated for gases, taken from ¼ of the 90x90 Mms. bars. Normal cooling and no heat treatment	Test piece of metal treated for gases taken from a rolled round 25 millimeters in diameter. Normal cooling and no heat treatment
Breaking Strain	40.5 Tons	44.76 Tons
Elastic Limit	27.0 Tons	35.5 Tons
Elongation Per Cent	28.9 Tons	24.4 Tons
Hardness	112	124
Resilience	17 31 Fibrous 19 Fracture 25	29 36 Fibrous 34 Fracture 34
Average	23	33.2

Here we see the ultimate strength, elastic limit, harndess (density), and resistance to fibrous fracture all materially improved in the self same metal by the simple elimination of occluded gases. The question as to whether resistance to corrosion is also improved is not considered.

Baraduc Miller recognizes the fact that the large volume of hydrogen eliminated was furnished by the decomposition of the moisture of the air used in blowing, during the conversion. To what extent this is a factor in the manufacture of open hearth steel, is uncertain, but that it is an ever present factor in the case of Bessemer steels, there can be no doubt. Baraduc Miller includes the following interesting discussion of the hydrogen content:

> "In the case of hydrogen, it is seen that the 5150 cubic meters of air injected into the converter contained, owing to the 5.672 grammes of water per cubic meter, 29,210.8 grammes of water capable of yielding, by complete dissociation, 3245.6 grammes of hydrogen, corresponding, at about 15° and under a pressure of 760 millimeters, with a volume of 36,058.6 liters.
>
> Now with 1098.7 liters of hydrogen given per ton of steel, measured at the ordinary temperature, the entire cast must have contained, at a given moment, 13,860.3 liters of hydrogen. It results, therefore, that the maximum amount of hydrogen

MICROSTRUCTURE OF TRANSVERSE SECTION

IRON NAILS OF ANCIENT ORIGIN
ABOUT 1400 YEARS OLD

Described by Sir Robert Hadfield in the 1912 "Journal of The Iron and Steel Institute."

fixed, at any rate momentarily, by the steel in some form or other, dissolved or in combination, must have been

$$\frac{13{,}860.5 \times 100}{36{,}058} = 38.5 \text{ per cent}$$

of the total volume of the available hydrogen.

> This throws an interesting insight into the extreme solubility of the gases, and in particular of the hydrogen, in liquid steel at a high temperature. It remains to ascertain if these gases are actually in solution or in combination, and also what is left of these gases in the steels at the moment of solidification in the ordinary conditions of manufacture."

The last sentence of this interesting quotation contains really the gist of the whole matter and applies quite as much to the question of nitrogen as to hydrogen. It has been pointed out in an earlier paragraph that gases may be held in steel either in a state of chemical combination or merely dissolved or occluded.

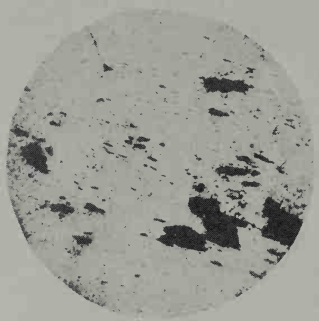
Up to the present stage of progress of our studies of the effect of gaseous impurities on the corrosion resistance of iron and steel, all the evidence so far in hand seems to point to nitrogen as a more important factor in respect to corrosion problems than the other gases under consideration. A vast number of analyses of all types of iron and steel have shown very significant differences in nitrogen content when taken in connection with the known behavior of certain metals under test and service.

In the discussion of the possible effect of nitrogen on the corrosion of metal, it must be borne in mind that the method used determines only the fixed or nitride nitrogen that is present, and tells us nothing about occluded or dissolved nitrogen. It must be understood, therefore, that such data as is here presented has to do only with the nitrogen which is held in the steel in combination as nitride.

Pure iron nitride has the theoretical composition Fe_2N, containing 11.8 per cent nitrogen. Commercial irons and steels as reported by a number of authorities and confirmed by numberless analyses which we have made, vary in nitride nitrogen content from traces in well made open hearth metals up to approximately .050 per cent in some Bessemer steels.

Although there is an abundant literature on the effect of gases and particularly nitrogen, on steel, there is very little evidence in regard to the quantity of free gas occluded in different types of metal.

MICROSTRUCTURE OF TRANSVERSE SECTION



IRON BILLHOOK OF ANCIENT ORIGIN ABOUT 1400 YEARS OLD

Described by Sir Robert Hadfield in the 1912 "Journal of The Iron and Steel Institute."

Austin electrically melted a number of samples of steel in a vacuum, collected and analyzed the evolved gas. One sample of mild open hearth yielded about one cubic centimeter of gas per gram, equal to about 35 cubic feet per ton of metal. The gas in Austin's experiments was pumped out in three stages, each of one half hour's duration. The electric current was first adjusted to raise the temperature of the test bar enclosed in a water cooled steel vacuum chamber to 900°C. The temperature was next increased toabo ut 1100°C, and finally in the third stage the bar was melted. The first extracts of gas from the mild open hearth steel analyzed as follows:

	Per Cent
Carbon Dioxide	7.7
Carbon Monoxide	18.4
Hydrogen	59.1
Nitrogen	14.8

Two other analyses of gas evolved during the last stage in the heating yielded:

	I	II
	Per Cent	Per Cent
Carbon Dioxide	1.4	1.5
Carbon Monoxide	65.7	59.4
Hydrogen and Nitrogen	32.9	39.5

The analysis of the open hearth steel given by Austin is:

Carbon	.09
Silicon	.05
Phosphorus	.05
Sulphur	.05
Manganese	.75

Another medium steel which evolved an even greater quantity of gas (1.35 cc. per gram) had the analysis:

Carbon	.49
Silicon	.35
Phosphorus	.02
Sulphur	.02
Manganese	.72

It is interesting to compare these results with those obtained by Baraduc Miller who, as described above, cooled basic Bessemer Steel in a vacuum.

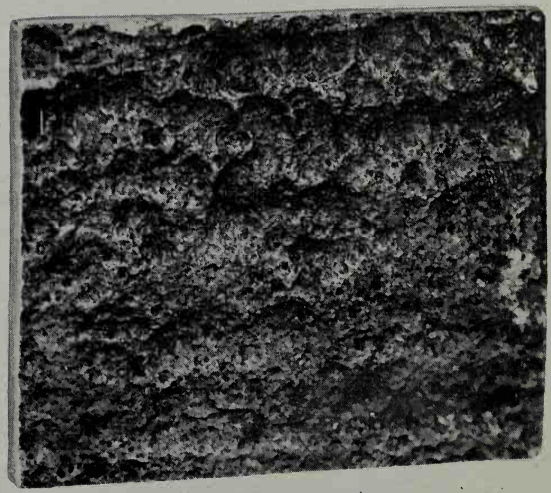
OLD IRON MERRIMAC GUNBOAT
TEST NO. 4211; FILE 28.

We have analyzed a sample of armor–plate taken from the Gunboat Merrimac which was sunk May 11, 1862.

ANALYSIS

Silicon	.038
Sulphur	.016
Phosphorus	.007
Carbon	.022
Manganese	.010
Copper	.018
Oxygen	.020

This material was a very high grade Iron, and was in a very good state of preservation after having been in the water for more than half a century.



Baraduc Miller obtained 75 cubic feet of gas per ton of metal, while Austin obtained in the case of the open hearth steel 35 cubic feet per ton. In the first case, however, it is presumable that some part of the gas collected would have escaped into the air, had it been allowed to cool in a normal manner.

In Austin's work, however, he started with a finished bar of cold steel, and it is difficult to escape the conclusion that all the gas evolved was actually held in some form or another in the body of the metal. That the gas was present in blowholes, is impossible, when we consider the volume relations which follow from Austin's experiments. Austin collected gas which, calculated to a tonnage basis, amounted, as has been shown, to about 35 cubic feet per ton of metal. Now, since a cubic foot of steel weight 480 lbs., there must have been a volume of gas present equal to approximately 7.5 cubic feet to each cubic foot of metal. This is a most extraordinary conclusion and one that will perhaps be unwelcome information to many steel users.

If the physical properties of steels of various types are affected by the nature and quantity of the occluded gases which they contained, it seems fair to inquire whether it might not be true that these same factors might exert an important influence on the much discussed question of corrosion resistance in relation to the chemical constitution of iron and steel.

The alleged good or bad effect of minute differences in the percentage composition of the metal has been so much discussed and argued about by a great number of investigators, that there is little left to be said or claimed in regard to the influence of these solid constituents. The effect of gas content has, however, been curiously overlooked in the discussion of corrosion problems heretofore, and yet it is probable that this one factor is the most important of all with relation to all the commercial metals, no matter whether we are considering a steel roofing sheet or a brass or bronze condenser tube.

Of the atmospheric corrosion tests consisting of many hundred full sized corrugated sheets, which have been described, exactly thirteen sheets out of about six hundred of the same age were found to have failed in periods ranging from 13 months to three years. It at once occurred to us that it would be interesting to select samples from ten of these failed sheets, and, after cleaning off the adherent rust with hydrochloric acid, have nitrogen and carbon monoxide determinations made, which would then be compared with similar analyses made from ten sheets taken at random, that were still in excellent

PURE IRON NAILS TAKEN FROM GRAVE OF SOLDIER AFTER BEING BURIED FOR 100 YEARS IN FORT ST. CLAIR CEMETERY, EATON, OHIO.

ANALYSIS OF NAILS

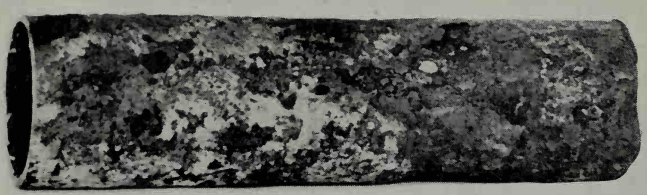
Sulphur	.007
Phosphorus	.100
Carbon	.020
Manganese	.004
Copper	trace
Silicon	trace
Oxygen	.031
Hydrogen	trace
Nitrogen	.008
Iron	99.830

condition on the test rack. Fortunately, in some instances strips of the original test sheets as they were before exposure had been preserved so that it was possible also to determine if any change of chemical composition of the base metal had occurred during the three years of outdoor exposure. This work was immediately put under way, with the following results:

Sheet No.	GOOD SHEETS		Sheet No.	BAD SHEETS	
	Carbon Monoxide	Nitrogen		Carbon Monoxide	Nitrogen
250	.003	.007	88	.011	.014
241	.005	.006	91	.016	.015
266	.006	.004	529	.012	.032
252	.003	.003	626	.016	.016
337	.009	.005	625	.024	.021
290	.016	.005	530	.014	.017
336	.011	.003	623	.013	.011
662	.019	.004	624	.021	.006
299	.014	.003	531	.015	.019
435	.012	.003	101	.027	.006

The bad sheets which failed, referred to in the above table, had all been purchased in the open market as examples of typical Bessemer steels, but the present point of interest in connection with them is the fact that they showed by analysis from two to five times the content of nitride nitrogen shown by the companion sheets which had not failed.

Taken in conjunction with the great difference in lasting quality of the two sets of sheets, the results are suggestive and significant. In the course of our experience a number of cases had been brought to our attention, in which steel in various kinds of service had failed in an extraordinary manner and in comparatively brief periods of time. Previous chemical analyses that had been made for the usual solid impurities, as well as microscopic examinations had not as usual revealed the reason for the sudden corrosion of the materials. In view of the significance of the results shown in the above table, however, it became a matter of interest to see what a complete analysis, including all gases, would indicate. The first case was that of a steel pipe which was perforated with holes after being buried for 14 months in an alkali soil. The second case is that of some railroad spikes that had corroded in an unusual and dangerous manner after being in use only a few years. An illustration of these failures is given. The results of the analysis of these failures is given in the following Table—and for the purpose of comparison the analysis of a very pure open hearth iron is also given:



FAILURE OF STEEL PIPE AFTER 14 MONTHS IN ALKALI SOIL DUE TO HIGH NITROGEN

ANALYSIS

Silicon	trace
Sulphur	.049
Phosphorus	.098
Carbon	.090
Manganese	.357
Copper	.014
Oxygen	.032
Hydrogen	trace
Nitrogen	**.041**
Iron	99.319

COMPARATIVE ANALYSIS OF STEEL AND ARMCO INGOT IRON

	Pipe Failure	Spike Failure	Armco Ingot Iron
Carbon	.090	.100	.010
Manganese	.357	.405	.025
Phosphorus	.098	.108	.004
Sulphur	.049	.041	.025
Silicon	trace	trace	trace
Copper	.014	trace	.040
Nitrogen	**.041**	**.033**	**.004**
Oxygen	.032	.082	.015
Carbon Monoxide	.052	.058	.015
Carbon Dioxide	trace	trace	trace
Hydrogen	trace	trace	trace

Other instances like the above, in which rapid failure has been accompanied by high nitride nitrogen content have been found, but as the present intention is rather to inquire into the subject than to arrive at final conclusions, a slightly different phase of the subject will now be referred to.

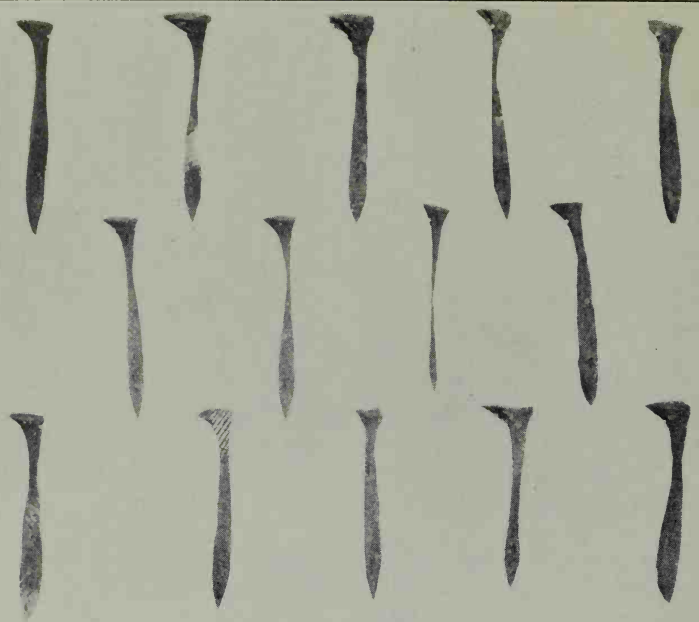
Among the examples of Bessemer steels included in the atmospheric exposure tests above referred to, Sheet No. 531 of Bessemer steel was perforated with numerous rust holes in 13 months, as was also sheet No. 88. Sheet No. 624 also of Bessemer steel did not fail until 33 months of exposure. The complete analyses of these three samples is given in the following table:

	Good Bessemer Sheet No. 624	Poor Bessemer Sheet No. 88	Poor Bessemer Sheet 531
Carbon	.010	.030	.015
Manganese	.480	.400	.310
Phosphorus	.082	.064	.060
Sulphur	.044	.031	.063
Copper	trace	.028	.010
Carbon Monoxide	.021	.011	.015
Oxygen	.055	.031	.071
Nitrogen	**.006**	**.014**	**.019**

In view of the great difference in lasting quality under atmospheric corrosion, these analyses are again suggestive. The nitride nitrogen of the two poor sheets is, however, from 2 to 3 times higher in the poor sheets than in the good sheet.

It has frequently been noted by a number of investigators that sheets that fail rapidly under atmospheric corrosion tests cover themselves with a much lighter colored and more loosely adherent rust than the more resistant sheets.

We heated small samples of iron and steel in an atmosphere of ammonia gas, and thereby raised the content of nitride nitrogen.



FAILURE OF STEEL RAILROAD SPIKES AFTER A FEW YEARS DUE TO HIGH NITROGEN

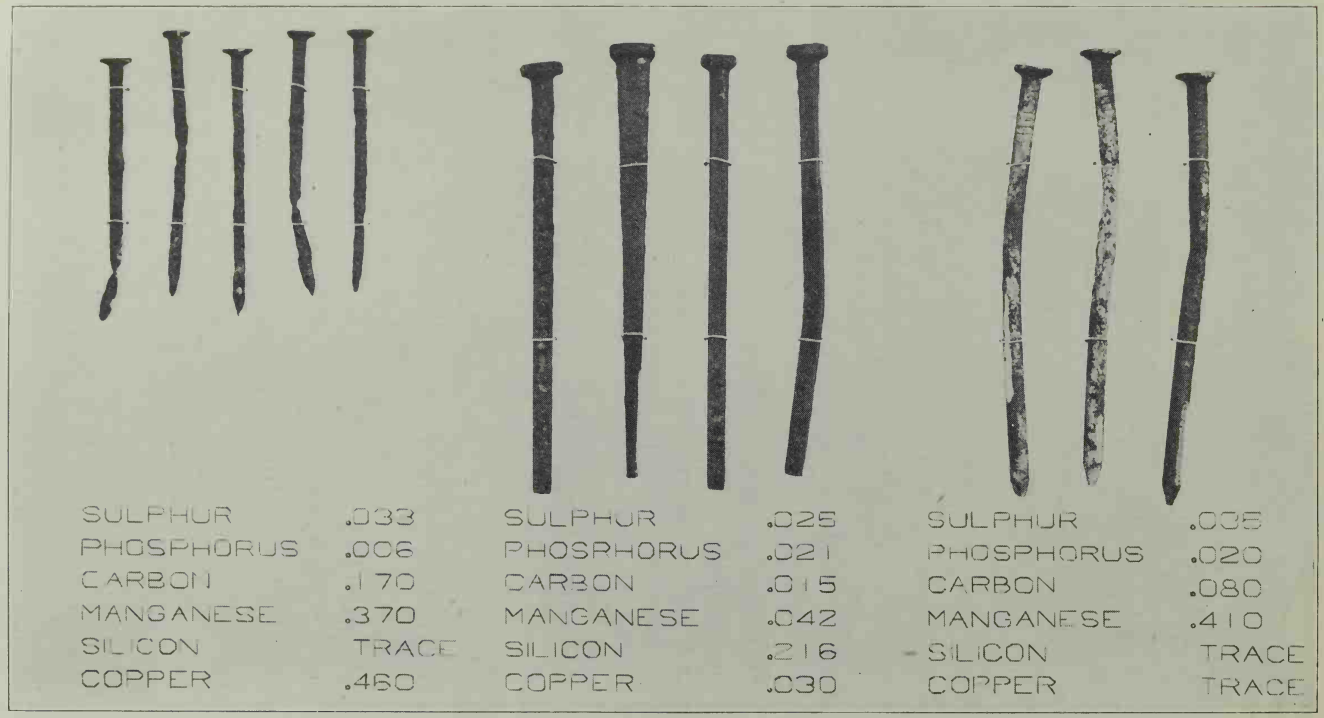
ANALYSIS

Silicon	trace
Sulphur	.041
Phosphorus	.108
Carbon	.100
Manganese	.405
Copper	trace
Oxygen	.082
Hydrogen	trace
Nitrogen	**.033**
Iron	99.231

In every case it has been found that with increased nitrogen content the attack of dilute acid on the specimen is stimulated. Two samples of pure iron were cut from the same sheet containing a normal content of nitride nitrogen of .003 per cent. One of these samples was heated in ammonia gas until the nitrogen had risen to .127 per cent. The two samples were then suspended side by side in 25 per cent sulphuric acid. The nitrified sample was destroyed in 6 days, while the untreated sample lasted 11 days. Unfortunately, no rational corrosion test is known, so that it has been impossible up to this time to test the corrosion resistance under atmospheric or various forms of service exposure, of test pieces that have had the nitrogen content increased by artificial means.

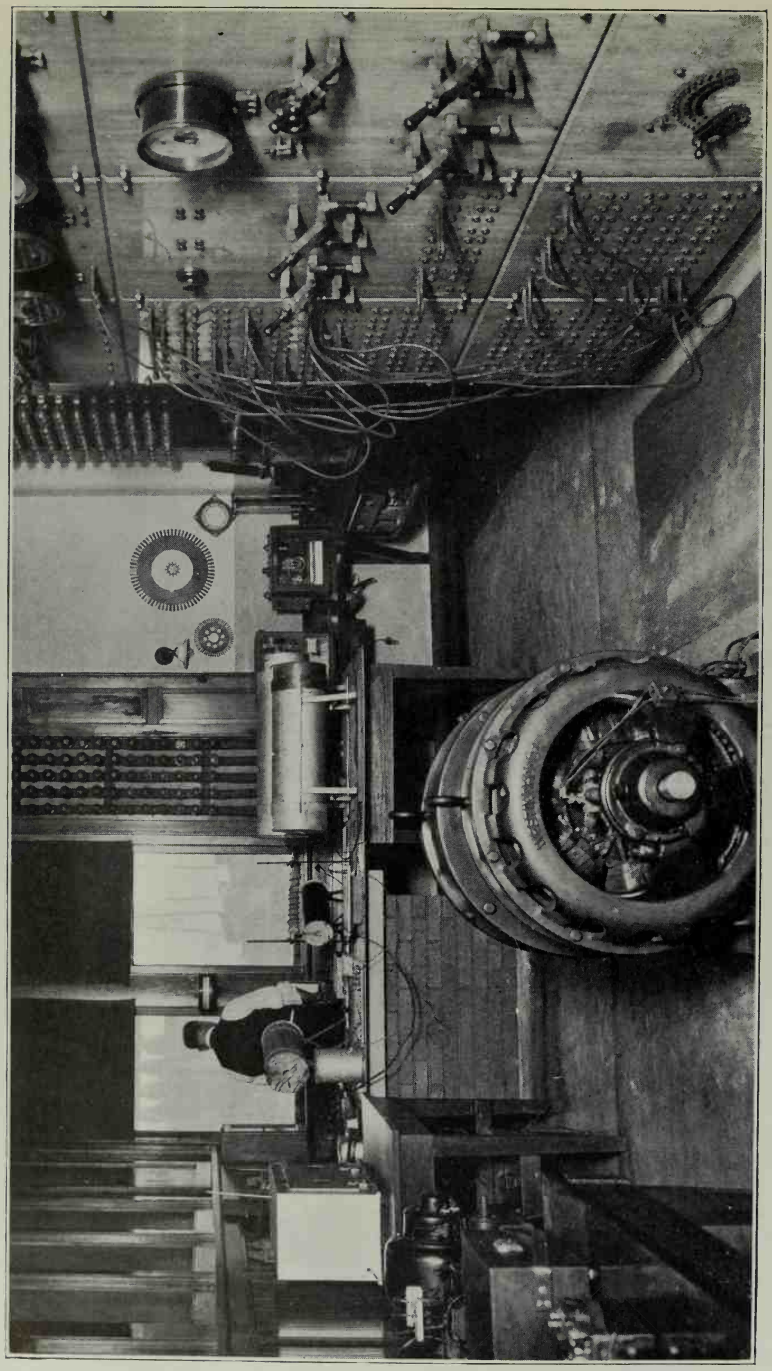
In the attempt to find an explanation of why high fixed nitrogen and high gas content stimulate rapid corrosion, we feel justified in stating that there is a considerable body of evidence which points in the direction of combined and occluded gas as being a very important factor in the corrosion resistance of various types of metal.

We have in our collections numerous examples of the old, hand forged, ancient irons that have shown very extraordinary resistance to corrosion under exposure, lasting in some cases for many centuries. It has always been a mystery, what gave these old irons their durability. With the repeated reheatings and hammering that these hand forged metals underwent, it is reasonable to suppose that they were wonderfully densified and degasified. They have always shown themselves to contain very low percentages of nitrogen.



All of these nails were driven in the same weir at Bakersfield, California and all were subject to the same corrosive influence of pure water The cut iron nails were in service for 29 years, the high copper steel nails and low copper steel nails were in service only 15 years.

MAGNETIC TESTING



View of pyrometer testing and apparatus to determine magnetic properties of electrical steel

MAGNETIC TESTING OF ELECTRIC SHEETS.

ELECTRICAL sheet steel products are today bought and sold on specification requiring careful tests for magnetic properties, both by the producer and consumer.

If the consumer does not have the facilities for magnetic testing he must rely upon the producer, and in this case it is well for him to know in detail what methods are being used.

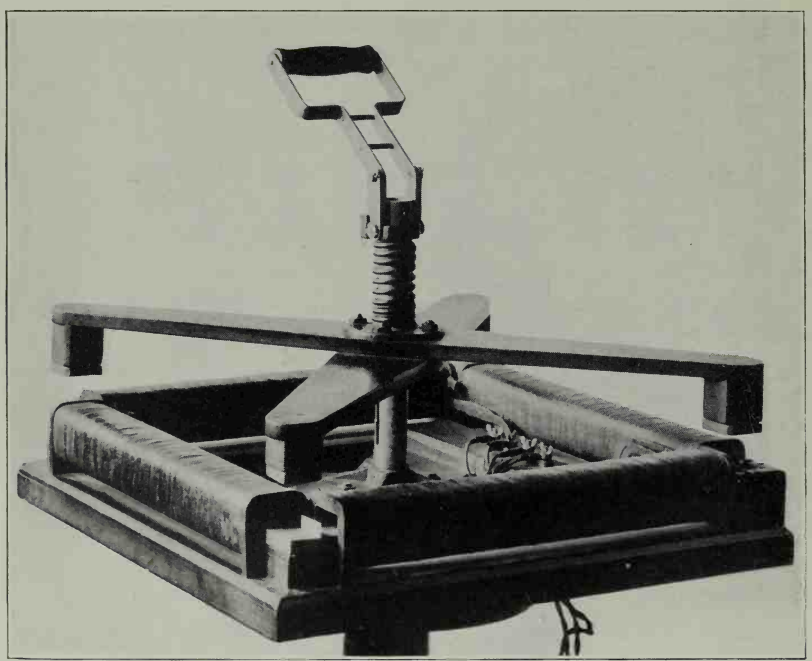
It is for the special benefit of this class of customers that we include the following paragraphs in this book. Others who are more advanced in magnetic testing will find the information useful in comparing results.

CORE LOSS TEST

The power consumption in electrical sheet steel, when subjected to an alternating magnetization is known as the core loss. The standard core loss is the total power in watts consumed in each kilogram of material at a temperature of 25 degrees C when subjected to a harmonically varying induction, having a maximum of 10,000 gausses and a frequency of 60 cycles per second. It is represented by the symbol W 10/60.

The method of test which we use is an outgrowth of that originally used by Professor Epstein and adopted by The American Society for Testing Materials in 1911. A photograph shows the arrangement of coils and sample pieces.

The magnetic circuit consists of 10 kg. (22 lbs.) of the test material, cut with a sharp shear into strips 50 cm. (19 11/16 ins.) long and 3 cm. (1 3-16 ins.) wide, half parallel and half at right angles to the direction of rolling, made up into four equal bundles, two containing material parallel and two containing material at right angles to the direction of rolling, and finally built into the four sides of a square with butt joints and opposite sides consisting of material cut in the same manner. No insulation other than the natural scale of the material (except in the case of scale-free material) shall be used between laminations, but the corner joints shall be separated by tough paper 0.01 cm. (0.004 in.) thick.



TESTING COILS—Epstein Core loss tests

The magnetizing winding consists of four solenoids surrounding the four sides of the magnetic circuit and joined in series. A secondary coil is used for energizing the voltmeter and the potential coil of the wattmeter.

These solenoids are wound on a form of any non-magnetic non-conducting material of the following dimensions:

Inside cross-section.....4 by 4 cm.
Thickness of wall.....not over 0.3 cm.
Winding length.....42 cm.

The primary winding on each solenoid consists of 150 turns of copper wire uniformly wound over the 42 cm. length. The total resistance of the magnetizing winding is between 0.3 and 0.5 ohm. The secondary winding of 150 turns of copper wire on each solenoid is similarly wound beneath the primary winding. Its resistance should not exceed 1 ohm.

A voltmeter and the voltage coil of a wattmeter are connected in a parallel to the terminals of the secondary winding of the apparatus. The current coil of the wattmeter is connected in series with the primary winding.

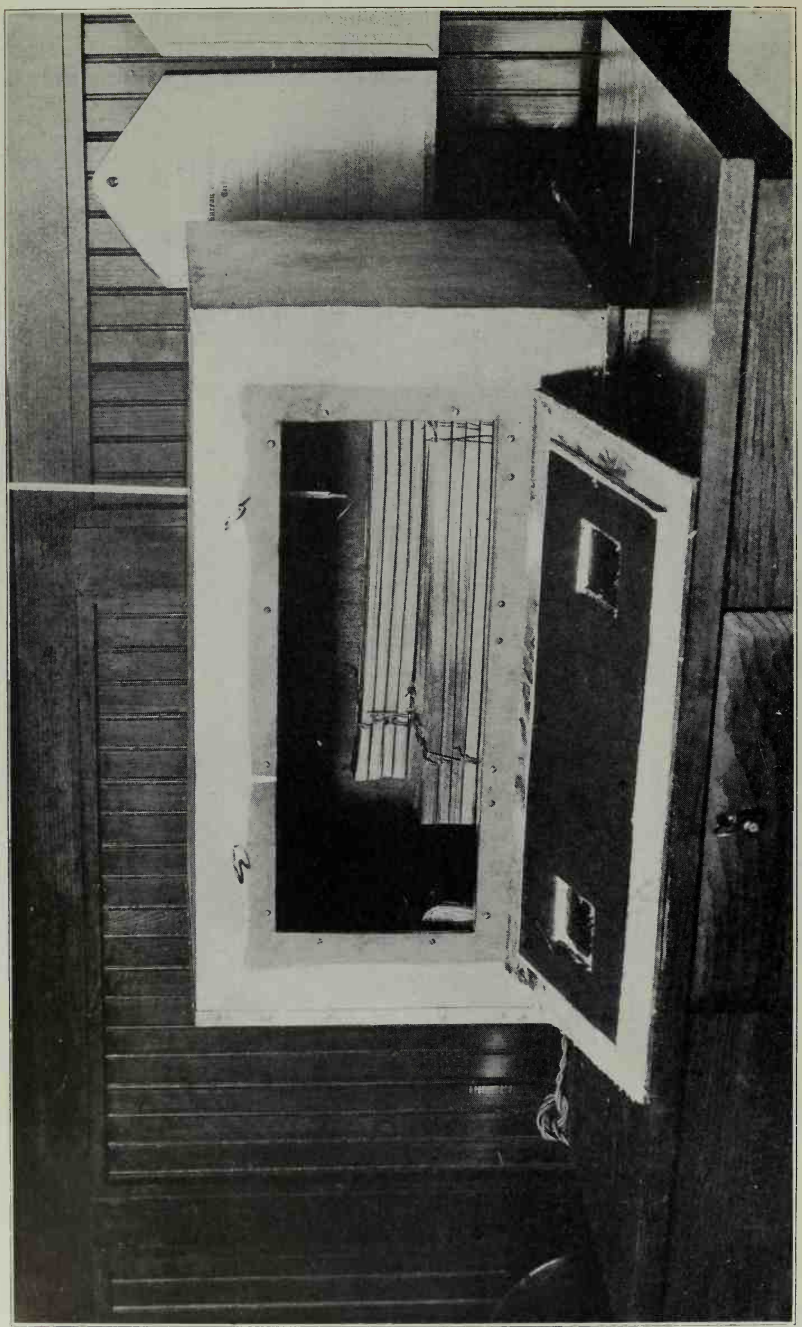
A sine wave electromotive force is applied to the primary winding and adjusted until the voltage of the secondary circuit is given by the equation:

$$E = \frac{4fNnBM}{41D108}$$

in which

f = form factor of primary E. M. F. = 1.11 for sine wave
N = number of secondary turns = 600
n number of cycles per second = 60
B = maximum induction = 10,000
M = total mass in grams = 10,000
l = length of strips in centimeters = 50
D = specific gravity = 7.5 for high-resistance steel
7.7 for low-resistance steel
E = 106.6 volts for high-resistance steel for sine voltage.
= 103.8 volts for low-resistance steel for sine voltage.

A specific gravity of 7.5 is assumed for all steels having a resistance of over 2 ohms per meter-gram, and 7.7 for all steels having a



Aging Oven for Magnetic Test Samples

resistance of less than 2 ohms per meter-gram. These steels are designated as high and low resistance steels, respectively.

The wattmeter gives the power consumed in the iron and the secondary circuit. The loss in the secondary circuit is given in terms of the total resistance and voltage. Subtracting this correction term from the total power gives the net power consumed in the steel as hysteresis and eddy current loss. Dividing this value by ten gives the core loss in watts per kilogram.

Sampling — From each annealing box of material we take sample sheets from top, center and bottom. Each sample representing not over 10,000 lbs. of material. If there is more than one heat of steel in the box the samples are so taken to represent each heat. All samples are recorded by box number, position of lot and heat number.

Procedure — 1. Cut the test material into strips 3 x 50 cm., half parallel and half at right angles to the direction of rolling.

2. Place on the balance a pile of strips weighing 2.5 kg. Add a second pile of the same kind, bringing the weight up to 5 kg. In each case the weight is taken to the nearest strip. Add in succession two piles of 2.5 kg. each, of the other kind of strips, bringing the weight up to 7.5 kg. and 10 kg. respectively.

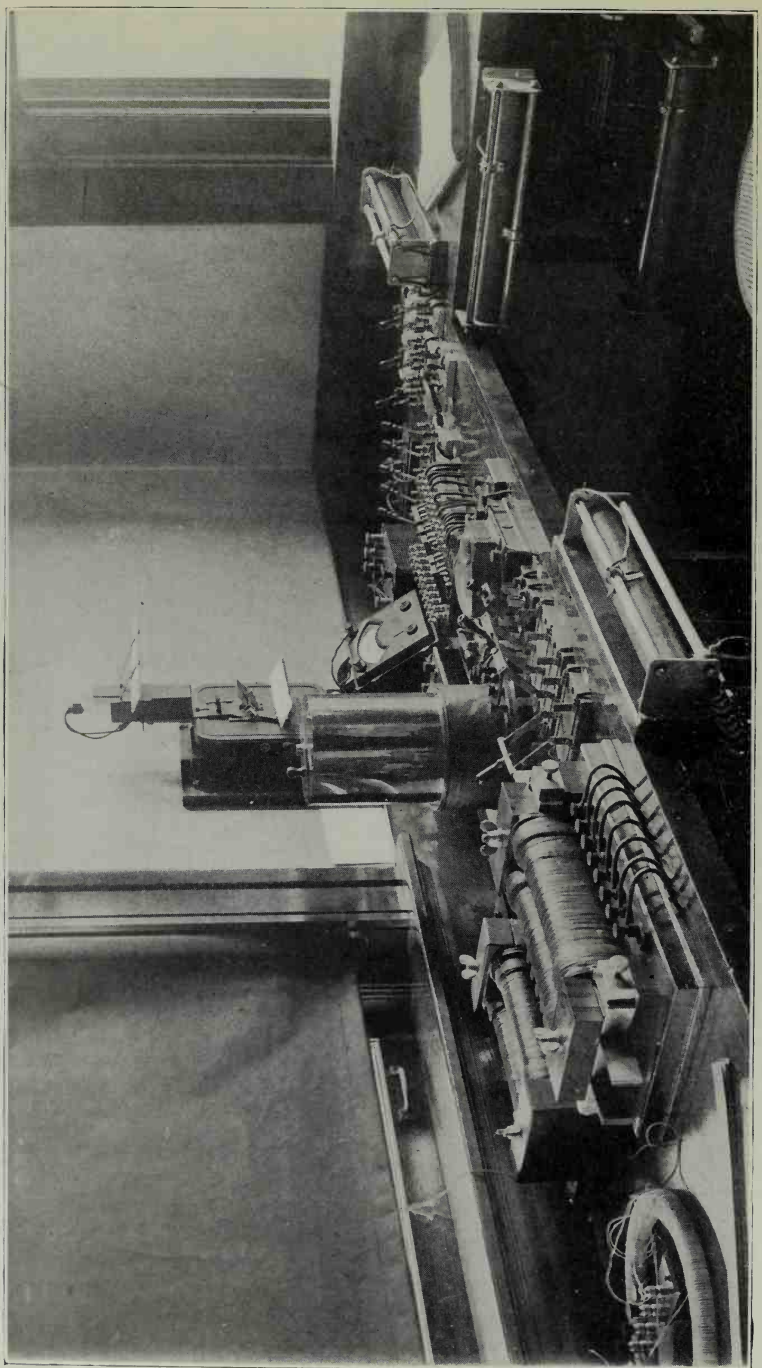
3. Secure each bundle by string or tape (not wire) and insert in the apparatus as indicated.

4. Apply the alternating voltage to the primary coil and tap the joints together until the current has a minimum value, as shown by an ammeter in series. Then clamp the corners firmly by some suitable device.

5. Shunt the ammeter and adjust the primary current until the voltmeter indicates the proper value. This adjustment may be made by an auto-transformer, by varying the field of the alternator, or by both, but not by the insertion of resistance or inductance in the primary circuit. Simultaneously the frequency must be adjusted to 60 cycles.

6. Read the wattmeter.

7. Calculations. Subtract from the wattmeter reading the instrument losses, which will be constant for any set of instruments and voltage, and divide by 10. The result is the standard core loss.



Burrows Apparatus for Measuring Permeability of Iron and Steel Sheets or Bars

AGING TESTS

Sheet steel after annealing is peculiar in its magnetic behavior and tends to go back to the unannealed state when continuously subjected to temperatures somewhat above normal room temperature. This increase in core loss is known as aging and is generally expressed as an aging coefficient in percent based on the initial core loss.

The standard test is based on a heating at 100 degrees C for 600 hours in a constant temperature oven such as shown in the accompanying photograph. Core loss tests to be made before and after the heating period.

PERMEABILITY TESTS

The above title is used in general to cover tests necessary to obtain data for normal induction curves or magnetization and saturation curves as they are sometimes called. From this data permeability values may be calculated and actual permeability curves plotted.

For this test we use the Burrows compensated double yoke method (described in the standard Hand-book for Electrical Engineers and also in Technical Paper No. 117 of the Bureau of Standards.) A photograph shows the apparatus as used in our laboratory.

The normal magnetic induction is the induction produced by a magnetizing force in a given piece of magnetic material which has been previously demagnetized and then subjected to many reversals of the given magnetizing force.

Both the induction B and the magnetization force H is expressed in terms of the C. G. S. electromagnetic unit (gauss).

The test material consist of 5 kg. of the strips cut as indicated for the standard core loss test.

The magnetic circuit is a rectangle having the test material for one pair of opposite sides, and the same or different material for the other pair, which may be shorter. The joints at each corner are alternately butt and lap, or may be clamped on the edges.

The magnetomotive force is applied in two sections. The main magnetizing coils consists of two equal and uniformly wound solenoids surrounding the test material. The compensating coils consist of two solenoids surrounding the yoke strips.

The test coil surrounds the middle portion of each bundle of test material. Four other test coils of half the number of turns are

View of room in Research Laboratory where permeability and conductivity tests are made on iron and steel

placed over the test material, approximately midway between the yokes and the center. The two center test coils are joined in series and the four end test coils are joined in series. The corresponding ballistic deflections, due to these two test coils, are measures of the magnetic fluxes through the underlying portions of the magnetic circuit. By connecting the two test coils so that the induced electromotive forces oppose each other, and adjusting the current through the compensating magnetizing coils so that there is no resulting ballistic deflection, an approximate uniformity of flux is secured through the greater portion of the test material, and the induction may be measured ballistically in the regular manner. The magnetizing force when the flux is adjusted to uniformity is that calculated from the uniform winding of the main magnetizing solenoids.

The cross-section of the magnetic circuit is determined as in the standard core loss test.

For curve work we obtain magnetizing force or H values corresponding to induction values "B" of from 2000 to 20,000 gausses by steps of 2000.

For obtaining permeability values at low and high inductions we determine "H" values for three values of "B" namely 6000, 10,000 and 16,000 gausses.

For routine commercial testing we find the above values very satisfactory for checking and comparisons.

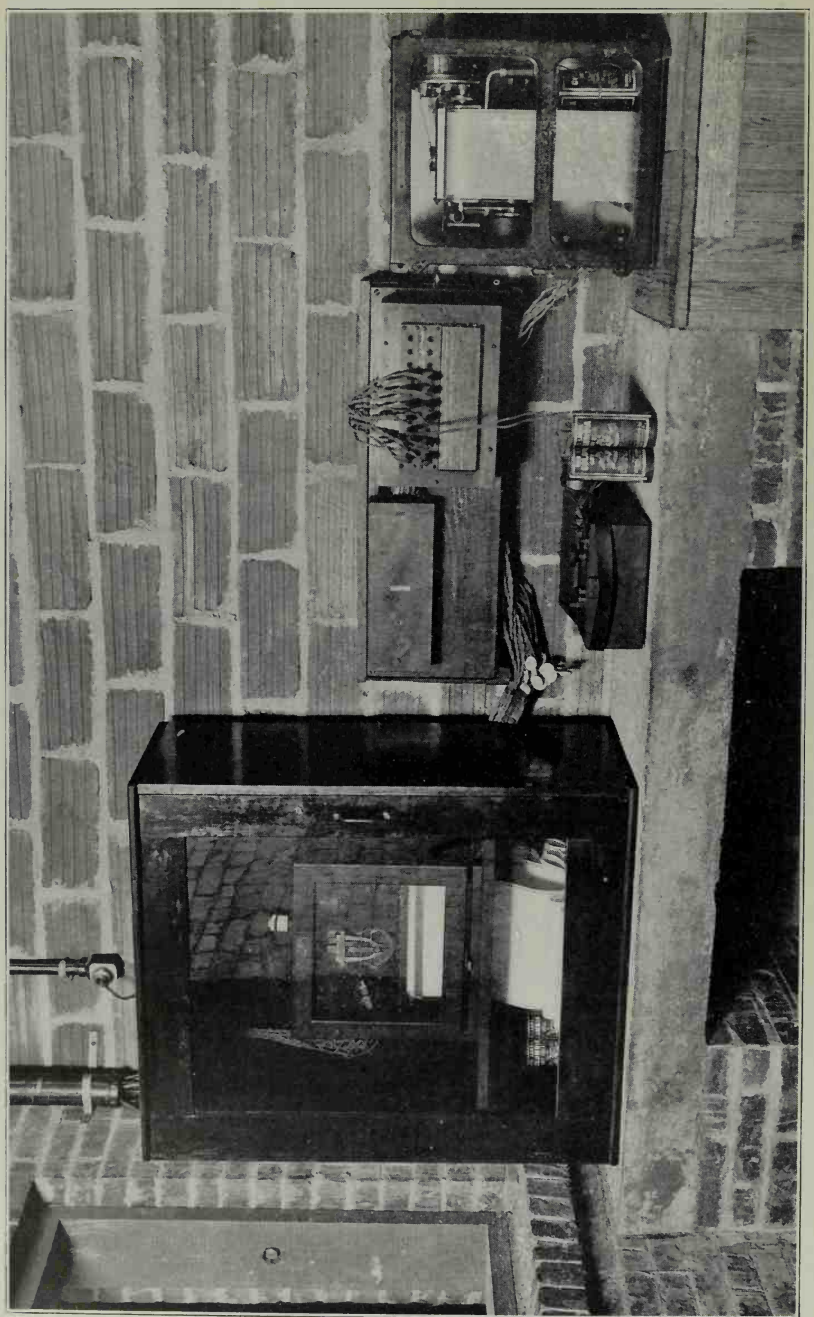
METALLURGICAL CONTROL

MICROSCOPICAL AND PHYSICAL TESTING

Pouring an experimental heat of 40 lbs. in the Research Laboratory.

METALLURGICAL CONTROL

IN addition to careful and constant chemical control the most exacting metallurgical supervision is necessary in the manufacture of "Armco" products. This supervision begins with the raw materials and follows through every operation to the finished product. A constant watch is kept upon the quality of the raw materials to insure that they are suitable to enter into the manufacture of Armco quality materials. In the Open Hearth Department in addition to the vigilant chemical control, a record is kept of the pouring temperatures of all heats and the greatest care is used to maintain uniform pouring temperatures. These temperatures are measured by means of optical pyrometers which will be described later. In the Blooming, Bar and Sheet Mills every care and precaution is used to see that the materials are fabricated in a manner to produce the highest quality products. The conditions of manufacture are watched continuously by the Operating and Research Departments to see that they remain constant and do not vary from Armco standards. In the Annealing Department where improper heat treatment can so readily produce undesirable properties in the sheets, a complete system of thermoelectric pyrometers is installed to record and to facilitate the control of annealing temperatures. In the Galvanizing Department thermoelectric pyrometers are also installed to safeguard the uniformity of manufacturing operations and thereby to insure the maintenance of Armco quality. In the Finishing Department the quality of the product is carefully tested and proved by microscopic and ductility tests as well as by thorough visual inspection. In fact throughout the whole mill every manufacturing operation is surrounded with every safeguard which experience and science can devise to insure the greatest uniformity and the highest quality in the finished products.



Pyrometer Equipment in Galvanizing Department

SCIENTIFIC HEAT TREATMENT

It is probable that the greatest advance which has been made in the metallurgy of iron and steel in recent years is the development of heat treatment upon a scientific basis. It has not been a great improvement in the art made at a single step by a single invention or discovery, as some of the improvements in the past, such as the Bessemer converter and the Siemens regenerative furnace. But scientific heat treatment has been a gradual development, the result of much painstaking investigation and research upon the part of many metallurgists. The fuller understanding of its principles and the broader application of them has resulted in higher quality in iron and steel products than was formerly thought possible.

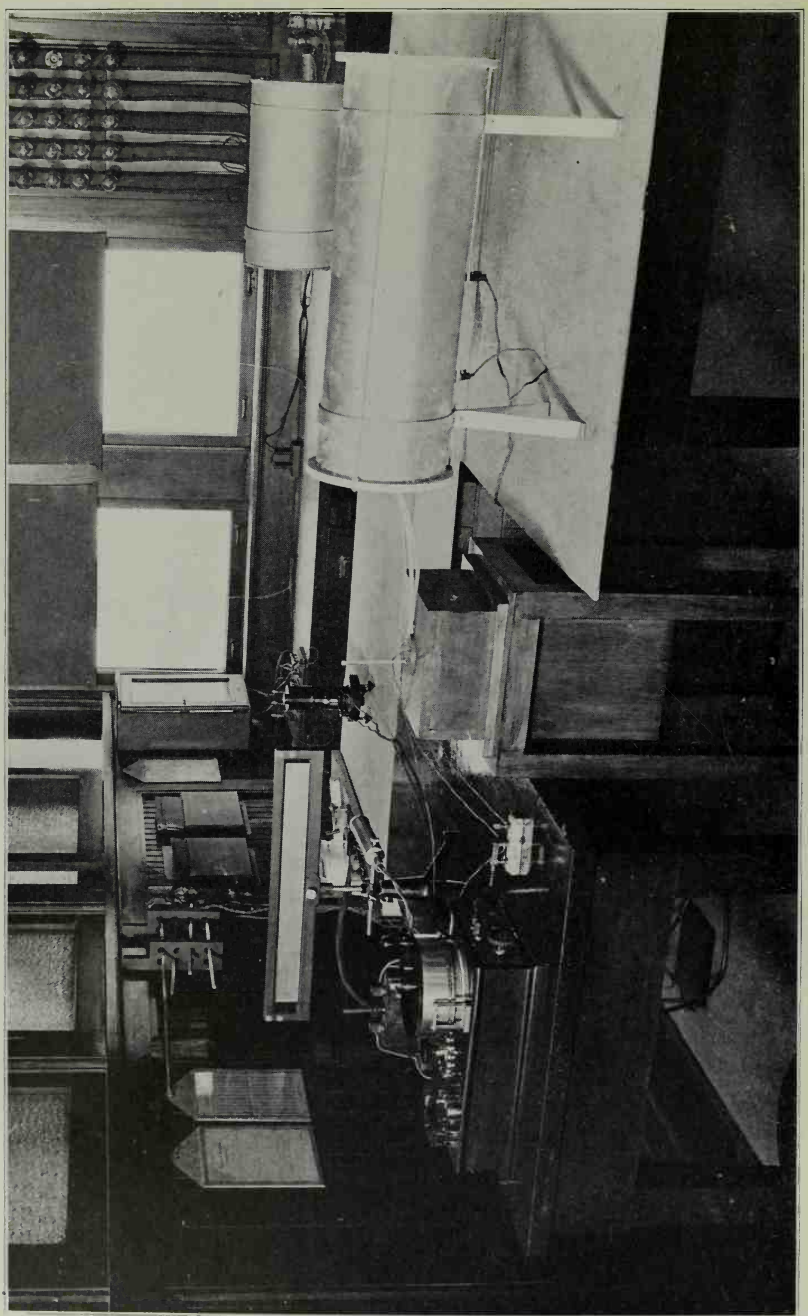
Heat treatment to the metallurgist means the application of heat or rather the manipulation of heat for the purpose of producing desired physical or structural properties in the material treated. Furthermore heat treatment is usually applied to finished or semi-finished material. In the forging or rolling of metal, stresses and strains are set up and only by heat treatment can they be removed. This heat treatment when done at the proper time and temperature, produces physical changes in the metal giving it the desired properties.

In the sheet metal industry this heat treatment is called annealing. It has been found that the length of time and the uniformity of the temperature are important factors. Improper annealing can ruin a sheet of metal for the purpose intended. It can make a soft sheet extremely brittle, yet re-annealing this material under proper conditions will bring it back again as ductile as before.

Especially in meeting the demands for a high finished or glossy sheet with deep drawing properties as demanded for automobile bodies and fenders, heat treatment has made a most important contribution to the industries of today.

The presence of strains in the sheets due to the rolling is also a contributing factor towards rapid corrosion and it has been found necessary to relieve such strains before coating as in the case of galvanized sheets which are to be used under exposure to the elements.

The narrow range between good and bad heat treatment can best be controlled by the use of metallurgical microscopes which reveal the grain structure for examination. Microscropic examination serves as a guide in obtaining a standard and uniform heat



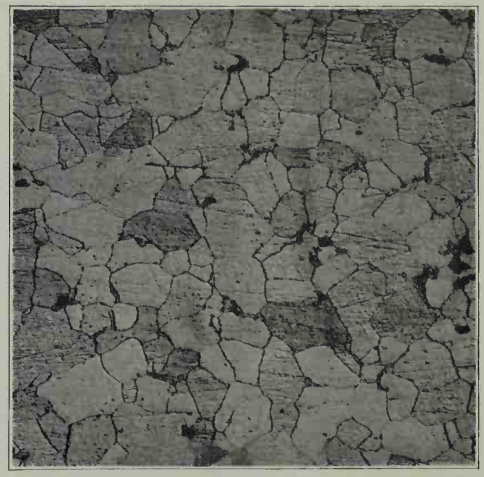
Laboratory Setup for Calibrating Thermocouples using Precision Potentiometer

treatment in the manufacture of the product. An example of the results of proper and improper annealing as detected by the microscope is shown on page 54. Both micrographs were taken from the same piece of steel; one after having been improperly annealed, and the other after having been properly annealed. The elongated grains and the strained condition of the metal is readily seen in the improperly annealed sample, and may be compared with the well-rounded equiaxed grains in the properly annealed sample.

Due to careful scientific research the organization of The American Rolling Mill Company has been able to produce many specialties in the iron and steel line. Notable among these are American Ingot Iron, Enameling Sheets, Automobile Sheets, Deep Drawing Sheets and sheets for electrical machinery, such as, transformers, motors and generators. In addition, billets are supplied from which wire is made with high electrical conductivity for use in telegraph and telephone lines. Billets are also furnished for the manufacture of welding rods and wire for the Electric or Acetylene Gas Welding of iron and steel plates or castings.

IMPROPERLY ANNEALED

PROPERLY ANNEALED



Micrographs of the same steel showing grain structure after improper and proper annealing

Carbon	Manganese	Phosphorus	Sulphur	Silicon
.10	.38	.010	.033	.033

MAGNIFICATION 100 DIAMETERS

THERMOELECTRIC AND OPTICAL PYROMETERS

The thermoelectric pyrometer equipment which The American Rolling Mill Company has adopted as standard for plant installations consists of base metal couples, and indicating and recording potentiometers for taking millivoltage readings. When necessary an alloy lead wire is used to carry the cold end to the potentiometer, where automatic correction for the cold end temperature is made.

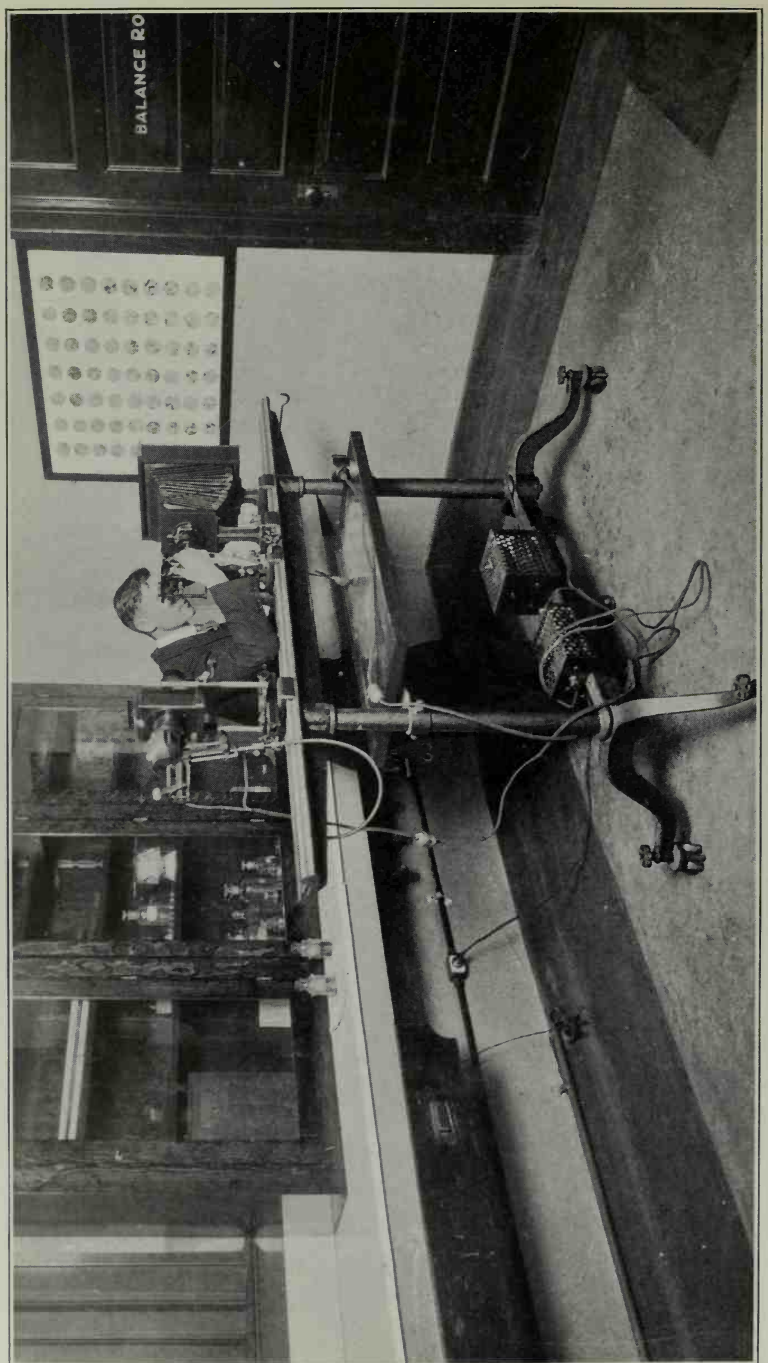
This base metal thermoelectric equipment is suitable for continuous service at all temperatures up to about 1600 degrees Fahrenheit. For temperatures above this, rare metal couples are used or another type of equipment, such as optical pyrometers.

All service instruments are checked at thirty day periods against a standard indicating potentiometer in the Research Department.

Thermocouples are checked and calibrated in the laboratory by comparison with standard platinum, platinum-rhodium couples which have been checked against a master couple calibrated by the Bureau of Standards at Washington.

Base metal couples which have been carefully checked in the laboratory are sometimes used as portable standards for checking in the mill.

Pouring temperatures in the Open Hearth Department, Soaking Pit temperatures and temperatures of hot ingots must be taken with a radiation or optical type of pyrometer. For this work we use an optical pyrometer. This instrument will measure temperatures from 1200 to 4000 degrees Fahrenheit.



Inverted metallurgical microscope in the Research Department, showing method of examining specimens

MICROSCOPIC TESTS

The Research Department is equipped with the latest and best equipment and accessories for the microscopic examination of metals. A photograph shows the inverted micro-metallograph used for this purpose. This is the equipment as perfected by Professor Sauveur of Harvard University, and manufactured by the Bausch and Lomb Optical Company. This inverted type of instrument is invaluable for Research work, enabling relatively large specimens to be supported on the stage and examined with ease.

The microscope has proved itself invaluable to the iron and steel metallurgist because of the information it discloses as to the physical structure of metals. Its extended use has been linked closely with the development of heat treatment for only by means of the microscope can the structural changes produced by heat treatment and annealing be observed.

Because of the close relation between annealing temperatures and grain size we have found the microscope to be a reliable index of the quality of the annealing, and a check by means of the microscope is made on every lot of sheets annealed. For this purpose one sheet is selected from near the top and the bottom of every annealing lot. A strip twelve (12) inches wide is cut from the end of each sheet and from the center of the freshly sheared edge of the strip we cut a small sample about $\frac{5}{8}''$ x $1\frac{1}{2}''$ for microscopic examination.

The sample is selected in this manner in order to have it representative, it having been proved by experience that a sample taken from this location is fairly representative of the whole sheet. The sample thus secured is polished, etched, and carefully examined and a record made of the quality of the annealing as indicated by the microstructure. No annealing lot which shows any indication of improper treatment is allowed to pass.

Four disc polishing table showing arrangements for polishing specimens for microscopical examination

Polishing Methods.

The specimen for microscopical examination is first ground on an emery wheel until the surface oxide, mat, etc., are removed and the specimen is flat. A four disc machine made by the University of Michigan is used to complete the polishing. The specimen is next ground on No. 000 emery cloth mounted on the first disc of the machine. Then the specimen is polished on the second, third, and fourth wheels to which is applied 3 F alundum, 65 C alundum and levigated alumina respectively, the specimen always being held so that the scratches run at right angles to those made by the preceding abrasive. Polishing is continued with each abrasive until the scratches made by the preceding one are removed.

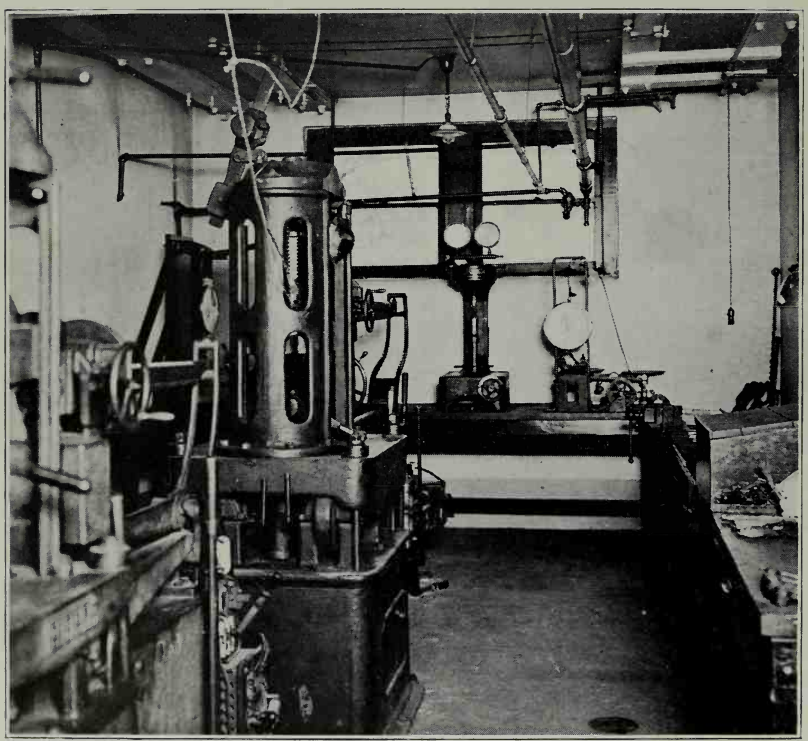
The abrasives with the proper amount of water are placed in ordinary 5-pint reagent bottles, fitted with 3-holed rubber stoppers. Compressed air for agitation is introduced through one hole, the syphon which conducts the water with abrasive in suspension to the polishing wheel passes through another, while the air is emitted from the bottle through the third one. The glass tube of the syphon is broken and about a 2″ section of rubber tubing inserted so that the amount of abrasive applied to the polishing disc may be regulated by means of a pinchcock.

Etching Methods.

Before a specimen is etched with any etching solution it is washed in ethel alcohol to remove any traces of oil or grease from the surface that might cause the etching to take place unevenly. After etching with the ordinary solutions the specimen is again washed in ethel alcohol to stop the action of the reagent and prevent staining or tarnishing. After washing, the specimen is dried with soft chamois which is kept free from minute particles of dust and grit.

For rapid etching when it is desired to examine only the shape and size of the grains an etching solution composed of 10 per cent nitric acid and 90 per cent ethel alcohol is used. A solution of 3 per cent nitric acid and 97 per cent ethel alcohol is generally used, and for ordinary purposes is very satisfactory and rapid.

When the specimen is to be examined minutely it is etched in a solution of 5 per cent picric acid and 95 per cent ethel alcohol.



General view in Physical Testing Section of Research Department

Picric acid as received contains 20 per cent water. Better results will be obtained if this water is removed. This may be accomplished by making a saturated solution of the picric acid in hot alcohol, allowing it to cool and recrystallize, and then filtering and drying. This is a more satisfactory method than attempting to drive off the water by heating, as the crystals fuse at 122° and explode at a slightly higher temperature.

For the identification of pearlite and segregated cementite in low carbon steel sheets the specimen is boiled 5 to 10 minutes in a sodium picrate solution. To prepare this solution proceed as follows:

Dissolve 25 grams of sodium hydroxide in 60 to 70 cc. of water, add 2 grams of picric acid, heat the solution until the picric acid is dissolved, and then bring the volume up to 100 cc. by adding more water. Before etching the solution should be brought to boiling. This reagent imparts a brown or blackish color to cementite.

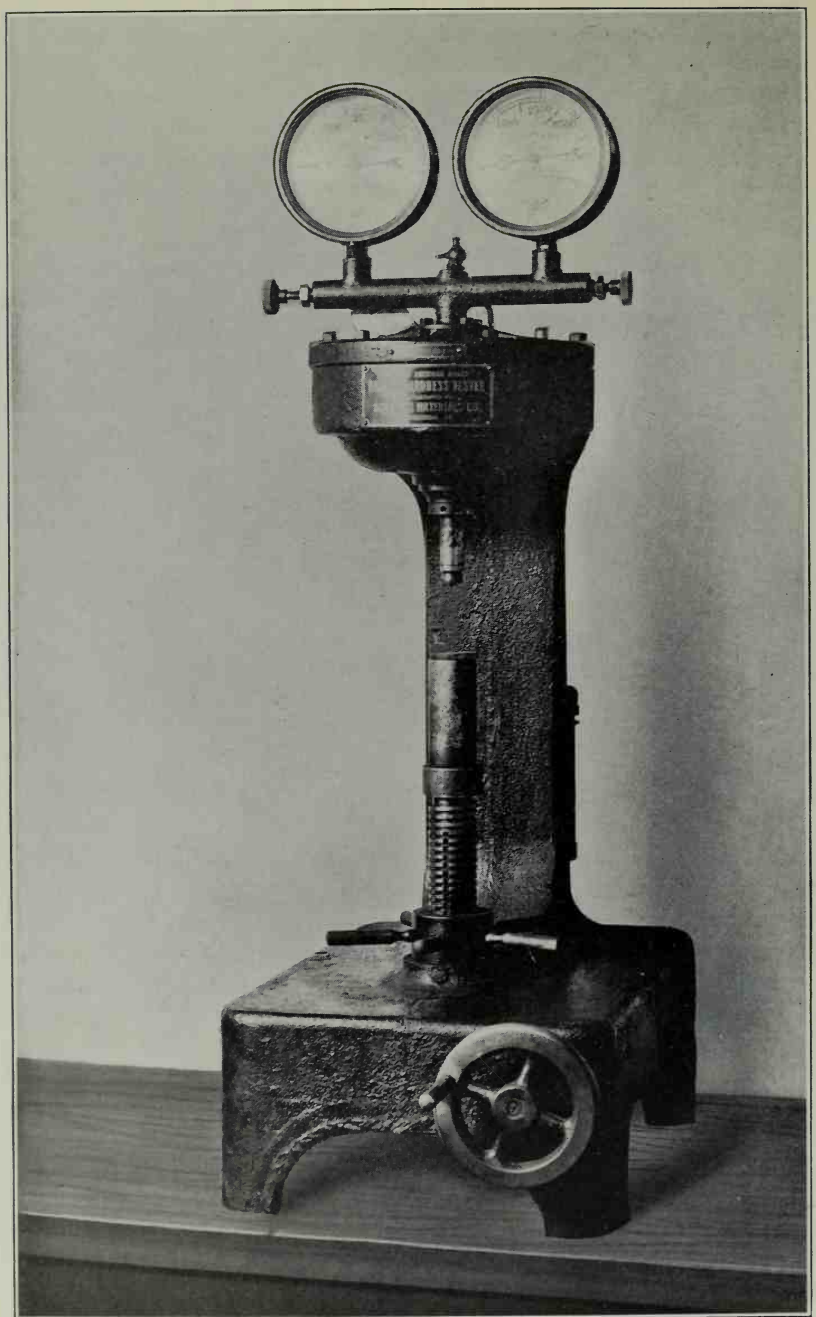
Special Methods.

In many cases heat-tinting affords valuable information when ordinary etching methods fail. Heat-tinting was originated by J. E. Stead and is described by him as follows:

"Heat-tinting consists in heating polished specimens of metals until their surfaces become colored by oxidation films.

"Alloys of iron and phosphorus, and commercial steel, contain part of their mass richer in phosphorus than other portions. In fact the iron and the phosphide are seldom intimately mixed in ordinary steel. When polished specimens are placed on the surface of a molten bath of tinman's solder[1], and the heat gradually raised, the portions of the specimens richest in phosphorus assume oxidation tints earlier than the purer parts; hence it follows that by the time the phosphorus-rich parts have passed through the transition stages of yellow-brown, to red and purple, the purer portions will have reached the yellow-brown or red stage, and if at this point the specimen be removed from the source of heat, the phosphorus-rich portions will appear under the microscope as purple or blue on a yellow-brown or red background. If the heating of the specimen be continued, the phosphorised part will assume a yellowish-white tint, while the purer part will reach the blue stage. Each part will pass through the complete range of color from yellow to blue and then to nearly white, but not at

1. An iron plate heated by a bunsen gas burner as suggested by Sauveur is used.



Brinell Hardness Testing Machine

the same time, because the purer portions always lag behind, the degree of lag depending on the variation in the proportions of phosphorus in the different parts.

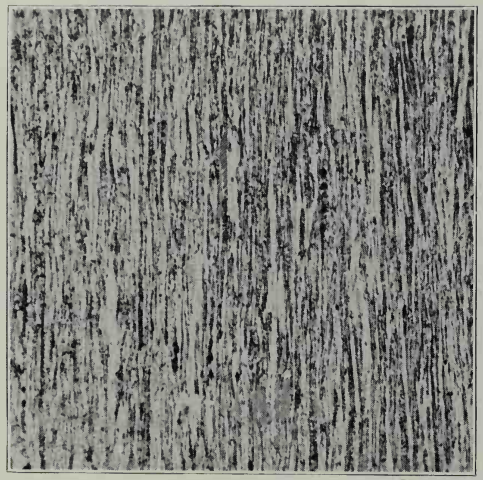
"Heat-tinting is also useful in intensifying the difference in color between ferrous sulphide and manganese sulphide when present together in steel. On heating polished metal containing inclusions of each sulphide until it appears to assume a uniform brown tint, the ferrous sulphide, which is naturally pale yellow, will be found under the microscope to have been colored purple, while the manganese sulphide, naturally a pale dove color, will have become white. If the heating be continued until the surrounding metal becomes blue, the ferrous sulphide will be blue and the manganese sulphide yellow.

"To obtain good results by heat-tinting, it is absolutely necessary first to apply to the surface a very dilute solution of some acid in alcohol. Picric acid answers admirably, but care must be taken to remove the solution employed before it has time to develop the pearlite or sensibly to etch the metal. After thoroughly washing the specimen in water, it is dried with a perfectly clean cloth and heated on a hot plate to about 150 deg. C. It is again rubbed with a warm clean cloth, and is then ready for heating to produce the color tint.

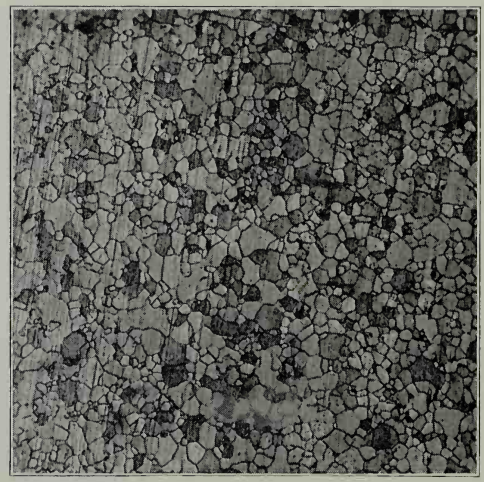
"It is difficult to explain why the preliminary acid treatment is necessary, but that it is so is proved in practice, for if it is omitted, the tinting is always unsatisfactory. It is possible that, during polishing, some of the softer metal becomes spread over the harder part, forming an exceedingly thin layer. This, however, is only a surmise."

In the course of many research investigations, recourse must be had to special methods of taking and preparing samples. Sometimes samples are so small it is impossible to hold them for polishing, in which case they are soldered to a large piece or mounted in solder in an ordinary pipe cap. Sometimes it is desired to polish and examine sheets on edge. In that case the samples are either soldered together or bolted with small stove volts. If bolted they are dipped in melted paraffin before polishing so that the paraffin will fill up the interstices between the sheets, which would otherwise give trouble when the specimen was etched.

INGOT IRON WIRE HARD DRAWN
BEFORE ANNEALING



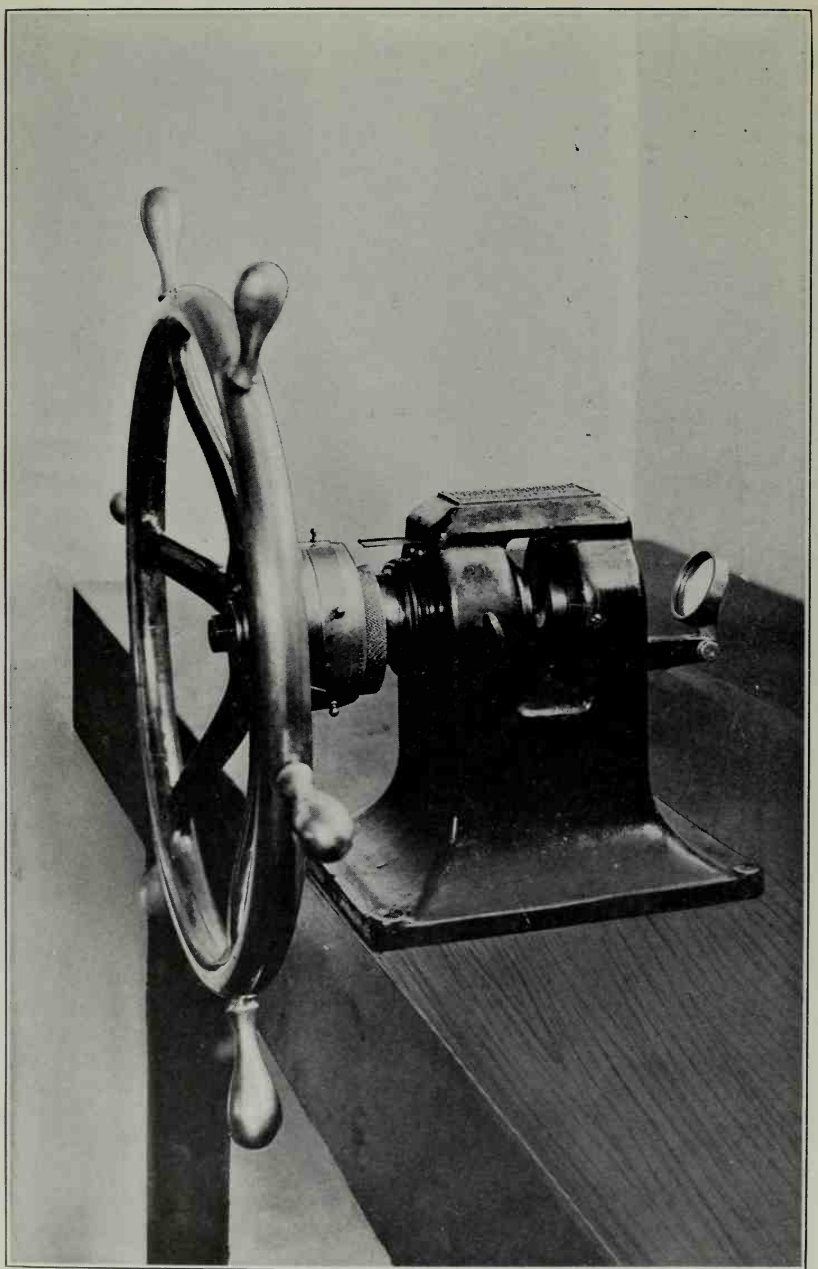
INGOT IRON WIRE
ANNEALED



Micrographs showing structures of Ingot Iron Wire in Hard Drawn and Annealed Conditions

MAGNIFICATION 100 DIAMETERS

Ofttimes it is desirable to use special etching reagents. For the use of these the reader is referred to the standard books on Metallography by A. Sauveur and H. M. Howe, and to the American Society for Testing Materials Standards for 1918, page 771.



Erichsen Draw Testing Machine for sheet metal

PHYSICAL TESTS

The Physical Testing Section of the Research Department is well equipped for carrying out all varieties of physical tests on iron and steel products as well as other materials when necessary. This equipment consists of a number of the more usual testing machines and several of more unusual ones. The following is a partial list of the equipment:

One 100,000 lb. Riehle Universal Testing Machine.

One 30,000 lb. Riehle Universal Testing Machine.

One Hydraulic Brinnell Hardness Testing Machine.

Several Erichsen Draw Testing Machines.

One Landgraf-Turner Alternating Stress Testing Machine.

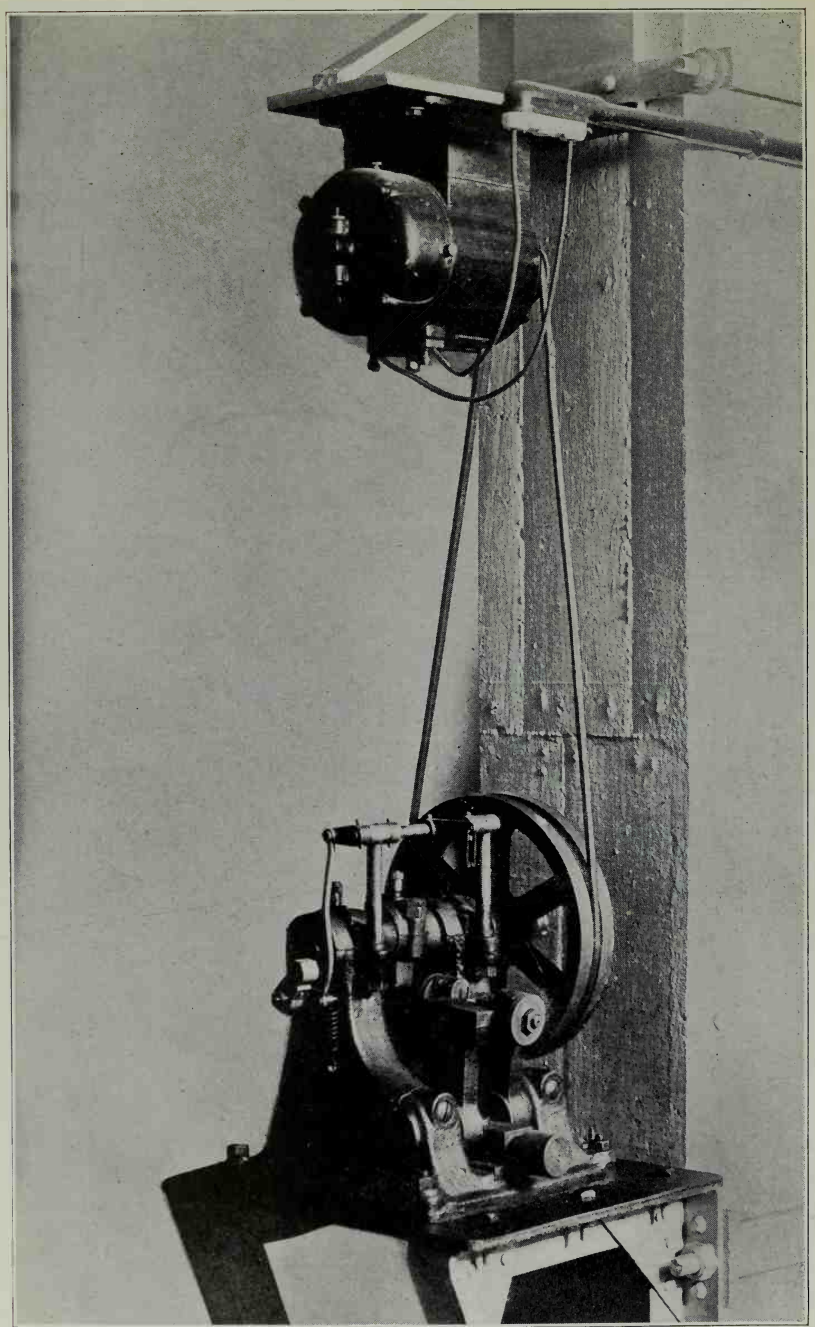
Several Bend Testing Machines, Scleroscope Hardness Testors, as well as several special testing machines designed for special purposes.

A general view of the Physical Testing Section is shown on page 60.

Tensile and Brinell Hardness Tests.

In making the usual Tensile and Brinell Hardness Tests, the standardized methods adopted by the American Society for Testing Materials are used. These methods are described in the 1918 Standards of the American Society for Testing Materials, page 759.

In addition to the usual tests many special tensile tests are carried out, among which may be cited tensile tests at high temperature. In these tests the specimen is tested while enclosed in an electrically heated tubular furnace which may be maintained at any desired temperature.



Landgraf—Turner Alternating Stress Testing Machine

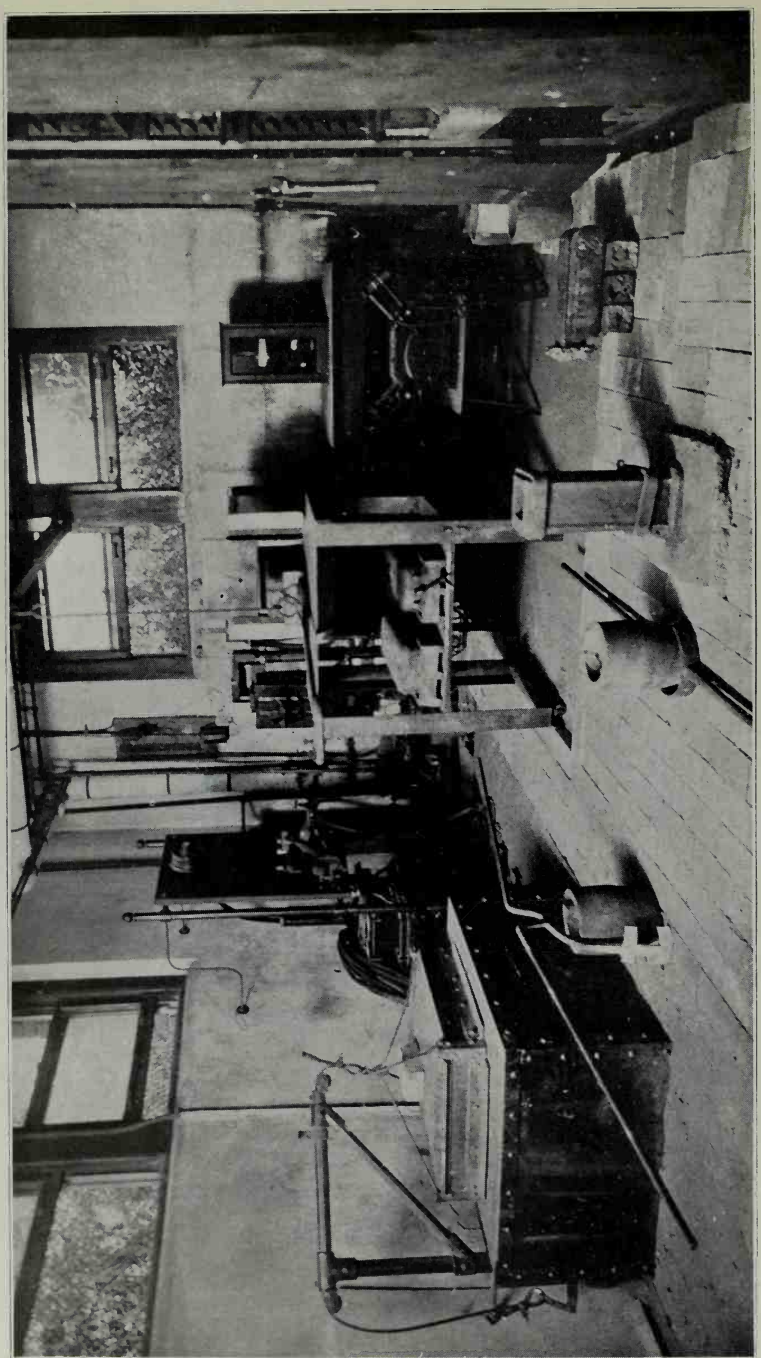
Ductility or Draw Test.

The ductility or draw test for sheet metal is made on the Erichsen Draw Testing Machine, a photograph of which is shown on page 66. This testing machine is used largely in research investigations in studying the drawing qualities of steel sheets and determining how that quality is affected by gauge, annealing temperature, grain size, and other factors.

When this machine is being used on a research investigation, samples for examination are taken from full size sheets in the same manner as described for the samples for micro-examination.

Alternating Stress and Impact Tests.

The value of dynamic tests as a means of determining the real value of materials for various engineering purposes where live loads are encounteerd is becoming more and more evident. While testing methods for determining dynamic strength have not as yet become stabilized and standard, the Physical Testing Section is, however, equipped with a Landgraf-Turner Alternating Stress Testing Machine, as well as impact machines of special design. With these machines the dynamic strength of materials under investigation is studied.



Experimental Electric Furnaces for melting and heat treatment.

THE EXPERIMENTAL FURNACE ROOM

Preparation of Alloys.

The Experimental Furnace Room is the section of the Research Department where the preparation of experimental alloys is carried out on a small scale, as well as annealing and heat treating experiments. The room is fully equipped with such furnaces as are necessary for this work, as well as pyrometers for accurate control of temperatures.

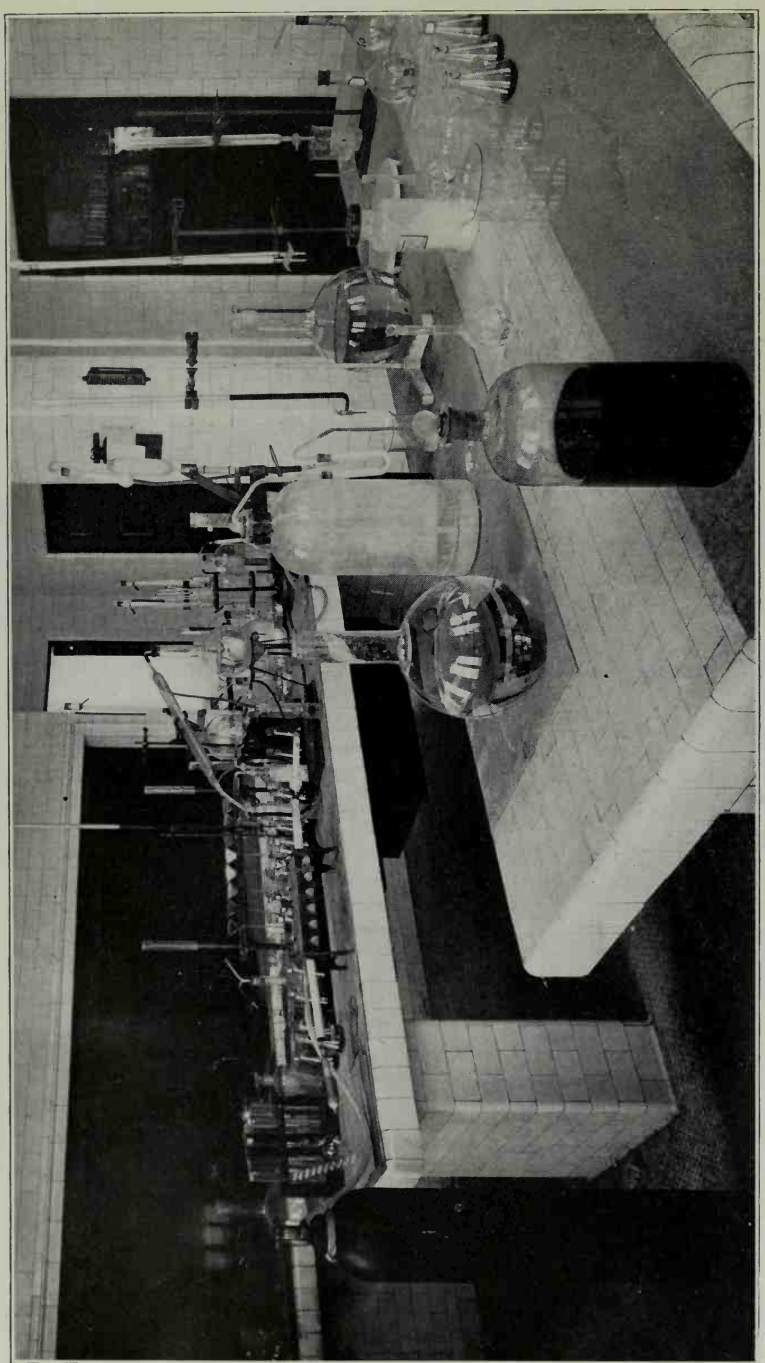
The furnace equipment which is all electrically heated consists of a Hoskins Crucible Melting Furnace of the carbon resistor plate type, a Hoskins Muffle Annealing Furnace, and a small arc melting furnace. The crucible melting furnace has an inside chamber dimension of 10″ x 10″ x 11″. It will conveniently accommodate a No. 18 graphite crucible and will melt without difficulty forty to fifty pounds of pure iron. The method of casting experimental melts is shown on page 48. It consists of pouring the molten metal from the crucible into a 3½-inch square split ingot mold. A forty-pound melt gives an ingot 3½ inches square by 12 to 14 inches long. With this furnace any alloy or series of alloys may be prepared in large enough quantities so that the physical, chemical, and magnetic properties may be determined. It is possible also to forge the small ingots into various shapes to determine the forgeability of the metal. The small ingots may even be rolled into sheets to determine the properties of the metal in sheet form.

Experimental Heat Treatment.

Experimental annealing and heat treating tests are carried out in the Hoskins Muffle Furnace. This furnace has a chamber dimension of 8″ high by 12″ wide by 26″ long. Temperatures up to 1800° F., can be readily obtained and steadily maintained. Small annealing covers and other auxiliary equipment for duplicating mill annealing conditions are available.

In all experimental furnace work accurate knowledge of temperatures is essential. The room is amply equipped with optical and thermo-electric pyrometers. Indicating and recording instruments are available and all are carefully checked against the laboratory standards whenever they are used.

CHEMICAL ANALYSIS



General chemical laboratory in Research Department, where our customers and also our own mill problems are studied

ALUMINUM.

KICHLINE[1] METHOD FOR THE DETERMINATION OF ALUMINUM OXIDE AND TOTAL ALUMINUM IN IRON AND STEEL.

IT has long been known that aluminum oxide when freshly precipitated is readily soluble in acids and when strongly ignited is very difficultly soluble in acids.

When metallic aluminum is added to steel while casting, its tendency is to unite at once with the oxygen existing in the steel, both as metallic oxides and as CO gas. The products of this reaction are Al_2O_3, the metals of the oxides reduced, and carbon. Since in regular practice there is only sufficient aluminum added to "quiet the steel," the aluminum added is nearly all converted to the oxide Al_2O_3. This aluminum oxide is, during the operation, heated to the temperature of pouring steel, about 1600 to 1650° C. (2912 to 3000 degrees F.), whereby it is rendered almost entirely insoluble in dilute hydrochloric acid.

In order to test the effect of high temperature on the solubility of Al_2O_3 the following experiment was carried out: Ten grams metallic aluminum were dissolved in hydrochloric acid, boiled low and replaced three times with nitric acid. This solution of aluminum nitrate was evaporated to dryness to drive off acid fumes; the residue was transferred to a platinum dish and placed into a muffle furnace where the temperature was gradually raised to a high heat, taking out a portion at intervals, and noting the temperature each time with a Scimatco optical pyrometer. Portions of aluminum oxide were removed at 815, 900, 980, 1065, and 1150 degrees C. Solubility tests were made by taking 1 gram of each portion and digesting for one hour with 100 cc. hydrochloric acid (1:1), filtering, and determining Al_2O_3 in the filtrate.

Portion heated to	815°C.	900°C.	980°C.	1065°C.	1150°C.
Gram soluble Al_2O_3	0.9317	0.3962	0.2337	0.0472	0.0390.

The increase in temperature was at the rate of 8 to 10 minutes between observations. Another portion of the same Al_2O_3 prepared above, was placed in a boat in the silica tube of an electric furnace and held for one hour at a temperature of 1000°C.; one gram of this

1 Metallurgical and Chemical Engineering, Dec. 1, 1915.

527

was treated as above with 1:1 hydrochloric acid and showed 0.0285 gram soluble Al_2O_3. One gram alundum, 120 mesh, was treated with hydrochloric acid as above and showed a mere trace of soluble Al_2O_3.

The solubility diminishes with increase of temperature and length of time to which the Al_2O_3 had been exposed to the heat. If Al_2O_3 exposed to 1000°C., will yield 2.85 per cent of its Al_2O_3 content, and alundum exposed to a little over 2000°C. yields a trace, it is safe to assume that the Al_2O_3 formed in molten steel would yield only 1 or 2 per cent of its content on treatment with dilute hydrochloric acid, which on such low figures as obtain with percentages of Al_2O_3 in steel is certainly negligible.

The above assertion has been borne out in practice, by adding just enough aluminum to deoxidize the steel, avoiding an excess. By employing the method as given below on such steels, all of the aluminum was found to be in the insoluble residue as oxide.

The method is as follows:

Dissolve 50 grams drillings in a mixture of 200 cc. strong hydrochloric acid and 300 cc. water at gentle heat, bring to a boil, allow insoluble matter to settle and filter through a double Baker & Adamson grade A paper or Schleicher & Schull No. 590, and wash with hot dilute (1:2) hydrochloric acid and hot water. Ignite the residue in a platinum fusion crucible. Add ½ gram pure sodium borate[2] calcined, and heat gently a few minutes till Al_2O_3 is in solution. Now add 5 grams pure sodium carbonate and fuse a few minutes longer until all is melted and in a state of quiet fusion. Dissolve fusion in hot water in a platinum or nickel dish, and determine Al_2O_3 in the usual manner.

In the filtrate the Al is directly precipitated as phosphate according to Wohler, as described by Blair. The precipitate is dissolved in HNO_3, and chromium oxidized by boiling with $KMnO_4$, and the Al precipitated from the Cr, Cu, etc., with ammonia. This precipitate is dissolved in HCl evaporated to dryness to separate SiO_2, and in the filtrate the Al is again precipitated as phosphate as before.

2 It is generally impossible to decompose all the Al_2O_3 by a sodium carbonate fusion alone.

DETERMINATION OF ARSENIC
DISTILLATION METHOD

Very good results can be obtained by the Distillation Method in determining arsenic in iron and steel. The methods used in the determination of this element require the use of either ferrous sulphate or cuprous chloride. We prefer the latter.

The method used in our laboratory has been carefully worked out and has been found to give very satisfactory results. The essential details being as follows:

Dissolve 20 grams of drillings in a 6 inch diameter casserole, using 300 cc. of nitric acid (1.20 Sp. Gr.). Heat slowly in order to prevent the reaction from becoming too violent. Evaporate to dryness on hot-plate and bake until no more nitrous fumes are evolved. Remove from hot plate and allow to cool, transfer the ferric oxide to distillation flask to which is attached a 50 cc. pipette which dips into a beaker containing 200 cc of distilled water. Place in distillation flask 40 grams of cuprous chloride and 300 cc. of concentrated hydrochloric acid, boil until about two-thirds of the hydrochloric acid has distilled over.

From the beginning of the distillation pass hydrogen sulphide gas into the distillate while same is being heated almost to the boiling point with the use of a small electric hot-plate. This will insure a rapid precipitation of arsenious sulphide which settles readily and can be easily filtered. After the distillation is discontinued remove the beaker containing the distillate from the source of heat, dilute to 500 cc. with cold water and continue to pass hydrogen sulphide gas into the solution until cold. Disconnect pipette and rinse inside and outside with two or three cc. of concentrated ammonia, allowing washings to run into beaker containing arsenious sulphide.

Allow the precipitate to settle and filter on asbestos using a Gooch crucible, wash with distilled water until free from acid. Transfer the asbestos felt containing the arsenious sulphide to a 250 cc. beaker. Add 10 cc. of fuming nitric acid or 10 cc. of nitric acid (1.42 Sp. Gr.) and 1 gram of potassium chlorate. Evaporate to dryness. Dissolve the arsenic acid in dilute hydrochloric acid, filter and wash with hot distilled water. Concentrate to about 20 cc., heat to boiling

and add 10 cc. of magnesia mixture, and 10 cc. of ammonia (0.95 Sp.Gr.) Continue to boil for 15 minutes, remove from the source of heat, add 20 cc. of alcohol and let stand for 5 hours. Filter and wash the precipitate on a weighed Gooch crucible, dry, ignite and weigh as magnesium pyroarsenate which contains 48.27% arsenic.

Magnesia Mixture

Dissolve 110 grams of crystallized magnesium chloride in a small amount of water. Add 140 grams of ammonium chloride, make distinctly ammoniacal with ammonia, and dilute to 2000 cc. with distilled water. Allow the solution to stand and siphon off the clear solution for use.

THE DETERMINATION OF BORON

After considerable experimenting with various methods suggested for the determination of boron in metallurgical products, we have found the following plan to be both accurate and rapid:

Fuse one-half gram of the powdered sample with potassium nitrate and sodium carbonate in a platinum crucible. Pour the melt upon an iron plate, transfer with the crucible to a porcelain dish and boil with just enough water to effect disintegration. Add a little sodium peroxide during the boiling to precipitate manganese. Filter into an Erlenmeyer flask and wash with hot water. Reserve the residue for manganese determination.

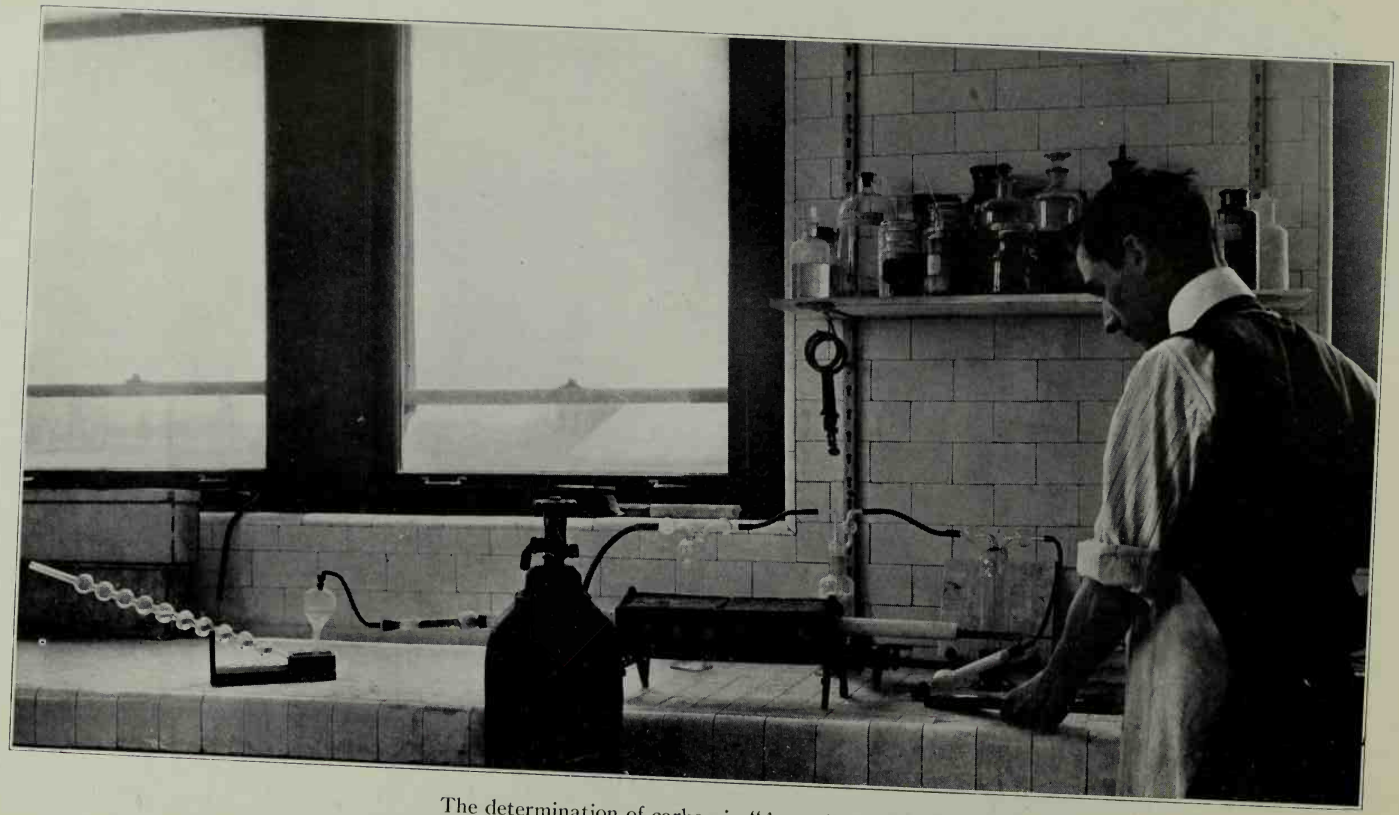
Acidify the filtrate with hydrochloric acid and then add a moderate excess of calcium carbonate. Connect the flask with an upright reflux condenser and boil about ten minutes to remove all carbon dioxide. Filter, cool to room temperature, add phenolphthalein and run in N/10 sodium hydrate to pink color; then add about one gram mannite. This destroys the color, and more sodium hydrate is added until the color is permanent, even on the addition of more mannite.

One cc. N/10 sodium hydrate equals .0011 gram boron. No deduction is required for blank.

One-half gram steel containing 4 per cent. manganese to which .03 gram boron was added in the form of fused boric acid gave by this method .0299 gram boron.

Dissolve residue from the fusion in hydrochloric acid, add 15 cc. sulphuric acid and evaporate to strong sulphuric fumes. Cool, dilute, heat till clear and make up to 250 cc. Take 50 cc. for manganese determination by the bismuthate method, and 50 to 100 cc for iron determination. Manganese may be determined on a separate sample if desired by the bismuthate method without separating the boron.

Silica is determined in the usual way, and carbon by direct ignition in oxygen, spreading the sample over ignited asbestos in the combustion boat.



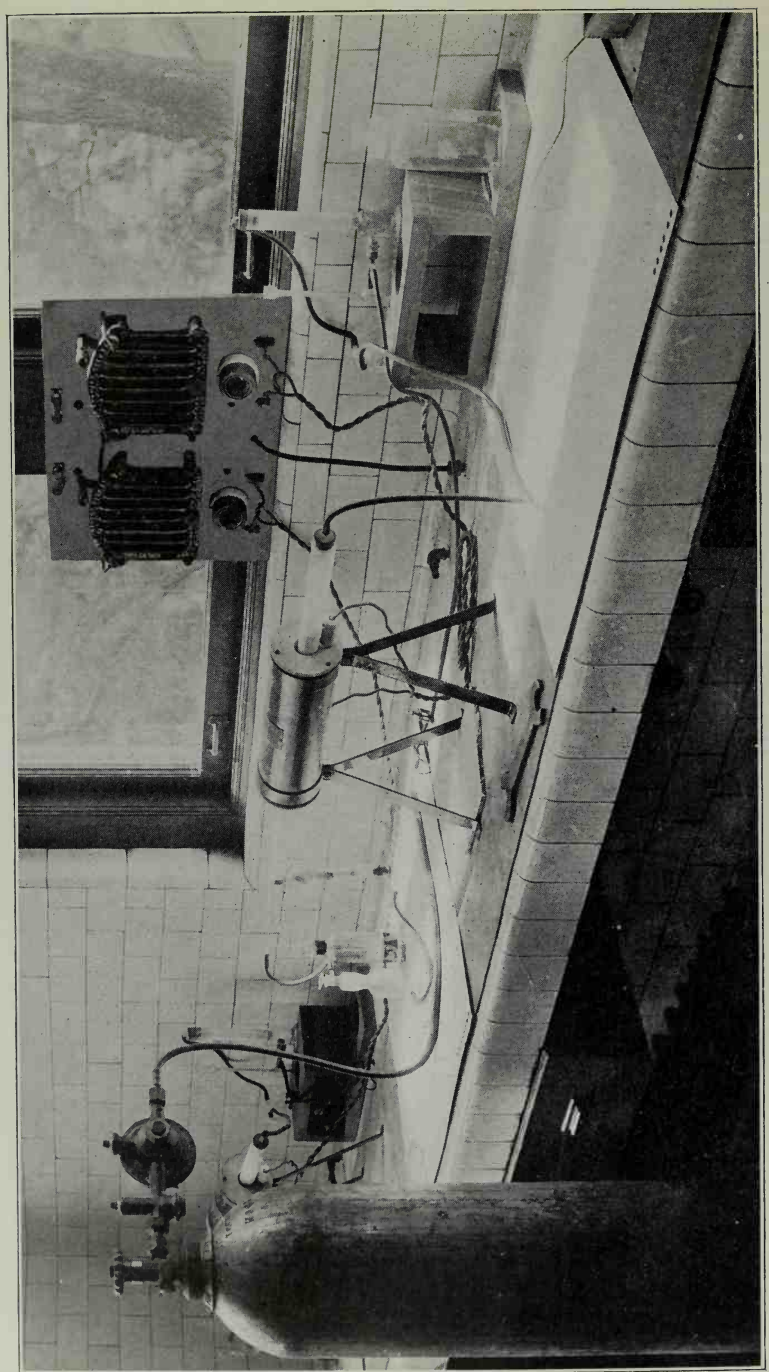
The determination of carbon in "Armco" Ingot Iron

DETERMINATION OF CARBON
COLORIMETRIC METHOD

In determining carbon by color it is essential that the standard contain approximately the same percentage of carbon as the sample, and also that the materials have had the same heat treatment. For the analysis of pure iron we furnish free a vial of standardized American Ingot Iron.

Dissolve .5 gram of the sample and .5 gram of the standard in 10 cc. of nitric acid, (1.18 Sp. Gr.), using 10 in. by 1 in. test tubes, heat over a Bunsen flame until the metal is dissolved and tubes are free from brown fumes. Cool gradually and pour into carbon comparison tubes, dilute standard with distilled water to at least 20 cc. (depending upon the carbon content) and add water to tube containing sample until colors match. The percentage of carbon present is determined by the following formula:

cc. of Standard : cc. of Sample :: percentage of Carbon in Standard : X.



View of carbon combustion room in Research Laboratory. This apparatus is used chiefly for the analysis of high carbon steel

DETERMINATION OF CARBON BY COMBUSTION

The determination of carbon in iron and steel is made by direct combustion, using a 7/8 inch bore x 30 inch silica tube heated in an electric furnace. A temperature of 1000 to 1030° C., is maintained with the use of a rheostat. The apparatus being standardized occasionally with the use of a platinum, platinum-rhodium platinum-rhodium thermo couple.

We have found that a gas furnace is not reliable where the gas pressure fluctuates considerably. This is due to the danger of overheating and causing devitrification of the silica tube, rendering it porous and the results obtained unreliable.

The following method has been found to be extremely accurate when dealing with traces of carbon such as exist in American Ingot Iron. The method we employ is essentially as follows:

Use a platinum or nickel boat approximately 6″x½″x½″, on which place a ⅛″ layer of 60 or 90-mesh alundum, free from carbon and alkalies, which has been heated to 1000° C., before being used for the first time. Make a channel in the alundum and place therein 4 grams of low carbon steel or American Ingot Iron, in the form of fine drillings. Spread compactly and evenly, then cover the borings with alundum.

Connect a Meyer bulb containing 75 cc. of clear barium hydroxide solution (30 grams $Ba(OH)_2 \cdot 8H_2O$, in 1 liter of water) direct with the silica tube.

Insert the boat containing the drillings into the central portion of the silica tube and quickly connect with the source of purified oxygen which is passed into the apparatus at the rate of not more than 100 cc. per minute, continuing the operation for 20 minutes. The flow of oxygen gas is regulated with the use of a high pressure reducing valve which will maintain a uniform rate of flow.

The Meyer bulb is disconnected, the solution filtered and washed with boiled distilled water. Care should be taken to filter under conditions which will prevent contamination by any carbon dioxide which may be formed within the laboratory.

The filter paper containing the barium carbonate is placed in a platinum crucible and ignited at a low temperature until all volatile matter has been driven off, and finish at a red heat until all carbon from the paper has been consumed.

The barium carbonate is weighed, multiplied by 6.08 and divided by the weight taken, which will give percentage of carbon.

For the analysis of high carbon steel we employ the rapid method in which we take 1½-grams, and absorb the carbon dioxide in soda lime contained in a Fleming bulb. A complete determination can be made in less than seven minutes.

IRON YARNING TOOL FOUND UNDER WATER MAINS AT CINCINNATI, OHIO. BURIED IN THE GROUND ABOUT 51 YEARS
TEST NO. 4524 FILE 103

Sulphur	**.030**
Phosphorus	**.030**
Carbon	**.015**
Manganese	**.112**
Copper	**.080**
Silicon	**.027**
Oxygen	**.050**
Nitrogen	**.004**

CARBON IN IRON [1]

By T. D. Yensen. [2]

It has long been known that carbon has a great influence upon the properties of iron and iron alloys. On account of the small quantities of carbon involved—0.1 per cent being regarded as a medium carbon steel—and the presence of carbonaceous matter in almost everything that is used in making chemical analyses, the correct determination of carbon in iron is the most difficult problem in iron and steel analysis. Not only are the total quantities of carbon very small, but carbon may exist in iron in various forms, one or more of which may influence the properties considerably, while others may have little or no influence upon these properties. The accurate determination of these small quantities and the separation of the different forms is, therefore, of great importance, and a large amount of work has been done lately to discover suitable methods to accomplish this purpose.

In another paper, Mr. C. J. Rottman is giving an account of the various methods that have been and are being used for the determination of carbon in iron and steel. He is also describing certain improvements in the method of analysis in connection with the combustion method—particularly in regard to absorbing the CO_2—improvements that greatly diminish the errors in the analysis. However, even these improvements were not considered sufficient. The method described in this paper is a combustion method, but it eliminates some of the most important sources of error inherent in the standard method, namely those due to absorption and weighing, and makes it possible to decide accurately whether all the carbon has been burnt out of the sample.

The standard method consists in heating the sample—in the form of shavings or chips—in a gas-tight tube to a temperature of 800 to 1000° C., passing oxygen through the tube and absorbing the resulting CO_2 in a bulb containing KOH or the equivalent. The increase in weight of the KOH bulb gives the weight of CO_2. Precautions are taken, of course, so that presumably nothing but the CO_2 from the sample is absorbed by the KOH bulb. As ordinarily practiced this method is satisfactory for carbon content of 0.1 per cent. or more, and if great care is exercised the error should be within ± 0.01 per cent. Mr. Rottmann states that with his improvements he is able to get an accuracy of ± 0.002 per cent. While this may be correct when the analysis is done with painstaking care, it is very

1 Transactions American Electro Chemical Society, April 1920.
2 Westinghouse Research Laboratory, East Pittsburgh, Pa.

doubtful if better than ± 0.01 can be obtained with the analysis in the hands of a regular analytical chemist.

Errors in the Present Method

The chief sources of error in the present method are:

1. Contamination of sample due to oil, grease, dust or dirt of any kind.

2. Dust or dirt or other carbonaceous matter in the combustion tube, in the sample holder, or in the connections between the tube and the rest of the apparatus.

3. Adsorbed CO or CO_2 in the walls of the combustion tube, or in the sample holder.

4. Admission of CO or CO_2 in opening the combustion tube.

5. Incomplete washing of the oxygen before it enters the combustion tube.

6. Incomplete oxydation of the carbon, resulting in some CO instead of all CO_2.

7. Incomplete absorption of CO_2 in the KOH bulb.

8. Weighing of the KOH bulb; moisture and dust collects on the bulb in uncertain amounts, and the weighing itself can at best be done with an accuracy of ±0.1 mg.

9. Carbon left in sample.

Some of these sources of error can be eliminated in the present method by careful manipulation, thus

(1) can largely be taken care of by careful sampling and boiling the sample in ether prior to placing it in the combustion boat.

(2) and (3) can be minimized by burning out the system, including the sample holder, with oxygen, prior to introducing the sample, while

(5) can be easily eliminated by means of active KOH and soda-lime in the train, according to standard practice.

This leaves (4), (6), (7), (8) and (9) as sources of error that can not readily be eliminated when the present method is used. The resulting errors vary to such an extent that it is difficult to get satisfactory blanks, and it is, therefore, necessary to make radical modifications.

Mr. Ryder's Results

Mr. H. M. Ryder has done a great deal of work on the elimination of gases from metals, including iron and iron-silicon alloys, by heating the samples in vacuo and analyzing the gases given off.[3] It was found that large quantities of CO and CO_2 were given off below 600° C., whereas additional CO was given off in varying amounts, depending upon the alloy, at and above the A_2 transformation point.

Based on these results it was concluded that the CO and CO_2 given off below 600° exist in the metal as adsorbed gases while the CO given off at and above A_2 is due to chemical reaction between the combined or graphitic carbon in the metal and the iron oxide present.

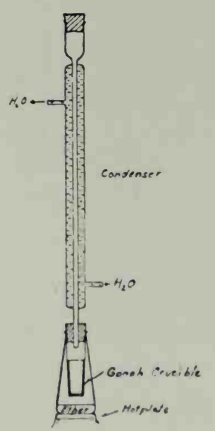
It is quite probable that these different forms of carbon, *i. e.*, that existing as adsorbed gases and that existing as combined or graphitic carbon, have different effects upon the physical properties of the metal and it is, therefore, of great importance to differentiate between them. This differentiation is taken care of in the new method as described below.

The New Method

A sketch of the apparatus is shown on Page 88, and is self explanatory. The following is a condensed statement of the procedure:

(*a*) *Preparation of the Sample*).

The sample is carefully collected to keep out foreign matter and the weighed portion then cleaned with ether in the apparatus shown on Page 87. The ether is evaporated in an Erlenmeyer flask and the vapor passed through the sample held in the Gooch crucible. The condensed vapor again passes through the sample on its way from the condenser to the bottom of the flask and is reheated by the rising vapors. The sample is thus exposed to a constant stream of hot, clean ether, carrying oily and greasy matter down into the bottom of the flask. This procedure should minimize source of error No. 1.



Apparatus for Cleaning Sample.

(*b*) *Preparation of Combustion Boat and Tube.*

The sample is then placed on a layer of specially prepared alundum in an alundum combustion boat that has previously been heated in the combustion tube to 1000° in a stream of oxygen. The sample is also covered with a layer of alundum. This precaution should eliminate

3 H. M. Ryder: "A Precision Method for the Determination of Gases in Metals." Trans. Am. Electrochem. Soc. (1918), 33, 197. "Analysis of Small Quantities of Gases." Jour. Am. Chem. Soc. (1918), 40, No. 11, 1656.

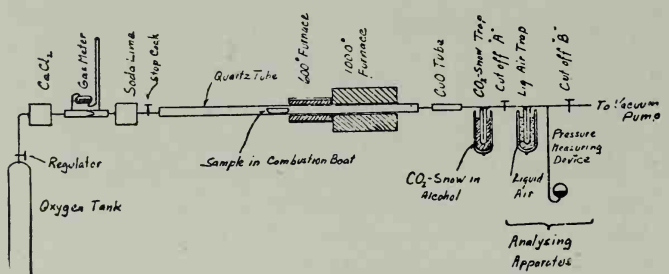
carbonaceous matter in the boat and in the combustion tube (sources of error No. 2 and No. 3).

(*c*) *Elimination of CO and CO_2 Introduced into Tube while Open.*

The combustion boat is now placed near the center of the tube with the furnaces moved over to one end, and the tube is closed. With the boat at room temperature or slightly above, the tube is evacuated to a pressure of 0.01 mm. Hg or better, eliminating all but traces of the free gases present in the system, thus eliminating source of error No. 4.

(*d*) *Determination of Adsorbed CO and CO_2.*

At the end of the above preliminary vacuum treatment, liquid air is placed on the trap and the 600° furnace is moved over the sample. The sample is treated at 600° in vacuum for 15 minutes or more, in order to remove the adsorbed gases. The CO_2 is "frozen out" in the liquid air trap and the amount determined by isolating the analyzing apparatus, allowing the CO_2 to evaporate, and noting the increase in pressure.



Apparatus for Carbon Determination.

(*e*) *Final Combustion.*

During the analysis of the CO_2 under (*d*), oxygen is admitted to the combustion tube up to atmospheric pressure through the $CaCl_2$ and soda-lime bulbs to wash it free from H_2O and CO_2, the flow being regulated by means of the stop cock and regulator. This filling of the combustion tube requires about 10 minutes, and at the end of this period the previous analysis is completed and the combustion tube is evacuated through the CO_2–snow and liquid air traps. When the pressure reaches 0.01 mm. Hg cut off *A* is closed, the system further evacuated to 0.001 mm. and the CO_2 analyzed as before, at the same time filling the combustion tube a second time with oxygen to make another analysis to make sure that all the carbon has been removed.

Complete combustion of the carbon is insured by passing the gases through CuO heated to 400°, eliminating source of error No. 6.

From the volume of the apparatus and the pressure, the amount of carbon can then be calculated. Absorption of CO_2 by KOH and weighing is thus done away with altogether, eliminating sources of error No. 7 and No. 8.

If there is any reason to believe that all the carbon has not been burnt during the previous combustion periods, the combustion process can be repeated any number of times until no further CO_2 is obtained. This can be done without introducing additional errors, which is not the case when the ordinary combustion method is used. Source of error No. 9 is thus eliminated.

Details of Apparatus

(*a*) *Furnaces.*

The 1000° furnace is a platinum-wound furnace with a porcelain tube 24 in. (60 cm.) long and 1¾ in. (4.4 cm.) bore, insulated with fire clay and sil-o-cel. It can be maintained at 1000° with an input of 1 kw., and at 1400° with 3 kw.

The 600° furnace is 12 in. (30 cm.) long, 1¾ in. (4.4 cm.) bore, consists of a silica tube wound with nichrome ribbon and insulated by means of magnesia pipe covering.

(*b*) *Quartz Tube.*

The quartz tube is 1⅜ in. (3.5 cm.) inside by 1⅝ in. (4.1 cm.) outside diameter by 6 ft. (180 cm.) long. One end is permanently sealed with a glass cap and cement, while the other end is provided with a ground glass joint.

(*c*) *The CuO Tube.*

The CuO tube is a Pyrex glass tube ⅝ in. (1.6 cm.) diameter, wound with asbestos and nichrome ribbon, without any heat insulation on the outside. It is filled with fine copper wire and heated to 400°.

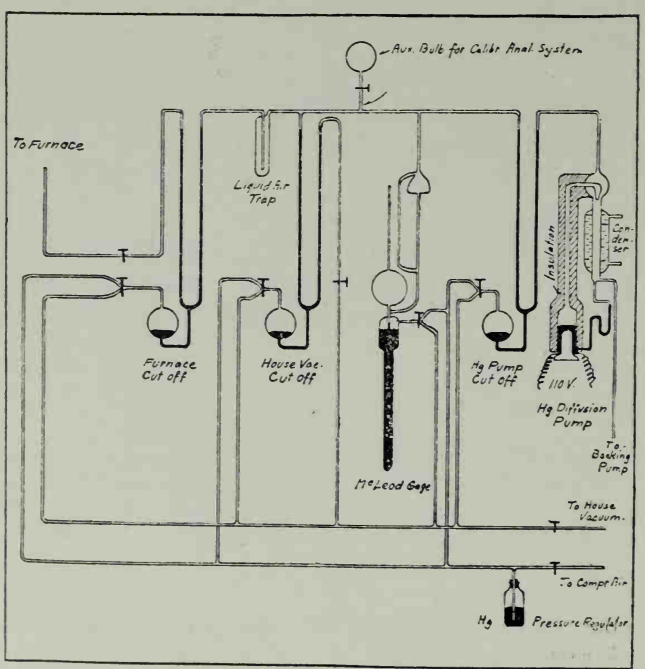
(*d*) *Connections.*

All connections between the different parts of the apparatus are made with sealed hard glass tubing, eliminating sources of error due to leaks.

(e) *Analyzing Apparatus.*

The analyzing apparatus was constructed in accordance with a design originated by Mr. Ryder. A sketch of same is shown on Page 90. The apparatus is made from hard glass throughout. The sketch shows the mercury in the cutoffs in position ready for analyzing the CO_2 frozen out in the liquid air trap. The volume then includes the trap, the bulb of the McLeod gage and the connecting tubing. Up to the zero points of the mercury column the volume is 259 cc. As the pressure increases, the mercury falls in the various cut-offs, thus increasing the volume of the system. The volume, being a function of the pressure, was consequently determined for various pressures by means of the auxiliary bulb. The relation between the pressure and the carbon is shown graphically on Page 91. Expressed in an equation this relationship is:

$C = (0.168 + 0.000095\ P)\ P$, where C = mg. of carbon and P = pressure in mm. Hg. Pressures up to 2 mm. are measured on the McLeod gage, while higher pressures are measured by means of the difference in level of the mercury columns of the mercury pump cut-off.



Analyzing Apparatus for Carbon Determination.

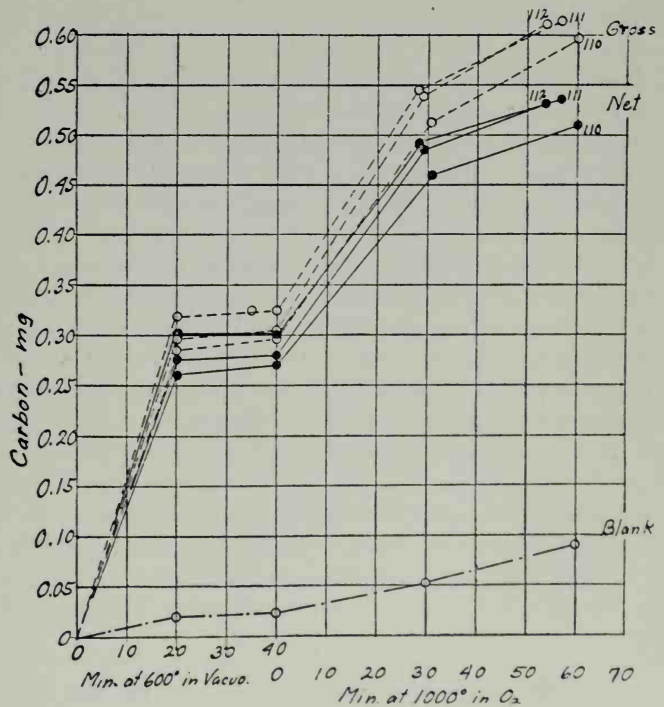
Pressures can be measured on the McLeod gage with an accuracy of ± 0.01 mm. Hg, and from the above equation it will be seen that this corresponds to a carbon content of ± 0.00168 mg. or ± 0.0000168 percent carbon if a 10 g. sample is used. This is far beyond the required accuracy, so that no difficulty will be had in eliminating sources of error No. 7 and No. 8.

(f) Diffusion Pump.

The pump was constructed in accordance with the design developed by Mr. J. E. Shrader,[4] and is of the type that can be operated at fairly high backing pressures. It is capable of evacuating the system down to 0.0001 mm. Hg in 5 minutes.

Preliminary Tests

Numerous preliminary tests were made to weed out the weak points of the apparatus and to determine the limits of accuracy.



Carbon in Electrolytic Iron. Standard Sample.

4 Westinghouse Research Laboratory. See Phys. Rev. N. S., Vol. 12, No. 1, July, 1918.

1. To determine the effectiveness of the various absorbing agents, a number of tests were made with ordinary air passing through properly prepared bulbs at a rate of 120 cc. per minute for 15 minutes, making a total of 1800 cc. The CO_2 escaping the first bulb was frozen out by liquid air in the analyzing system and the amount determined as usual. The result is shown in Table I.

Table I

Efficiency of Various Absorbing Agents for CO_2

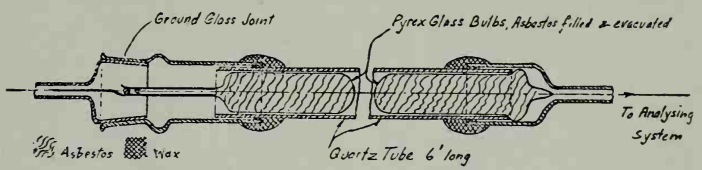
No.	Absorbing Agent	Amount CO_2 in 1800 c.c. Air not Absorbed by Absorb. Agent mg.	Efficiency of Absorb. Agent Percent
1.	None	0.840	0
2.	KOH and Ca Cl_2	0.350	60
3.	Fleming Bulb (Soda lime)	0.025	97
4.	Liquid Air, Plain Trap	0.025	97
5.	Same, Trap filled with Glass Wool	0.001	99.9
6.	Same, Trap filled with Steel Wool	0.001	99 9

This shows that the rate used was too high for the KOH bulb but safe for the Fleming bulb. The plain liquid air trap passed 3 percent of the CO_2, while the traps filled with steel or glass wool passed only 0.1 percent of the total CO_2. Hence the adoption of the glass wool trap for the analyzing system.

2. A glass wool filled liquid air trap was at first also used in the purifying train, but it was found that the suction of this trap on the CO_2 given off in the furnace during the passage of this oxygen was sufficient to freeze out from 10 to 30 percent of the CO_2 given off. Furthermore, the liquid air trap in the purifying train necessitated passing oxygen through the furnace and the trap at a reduced pressure in order to keep the oxygen from liquifying in the trap. These considerations led to the adoption of the Fleming bulb for the purifying train. Table I shows that this bulb allows only 3 percent of the total CO_2 in the oxygen to pass through, and as the amount of CO_2 in the oxygen is very small (only 0.02 mg. per 1800 c. c. as compared to 0.84 mg. for air) it is evident that the Fleming bulb is sufficiently effective.

3. Asbestos plugs were originally used at the exit end of the combustion tube to protect the wax seal from undue heating and no protection was used at all at the entrance end, where De Kotinsky

cement was used to seal the glass to the quartz tube. It was soon found however that this arrangement caused a great deal of trouble, and the arrangement as finally adopted is shown on Page 93. The protecting bulbs at the ends consist of Pyrex glass filled with asbestos, evacuated and sealed off. They are securely held in place so that they cannot be drawn into the heated portion of the tube. They prevent heating of the wax very effectively without the use of materials in the combustion tube that absorb and give off gases in considerable and uncertain amounts.



Combustion Tubes.

4. Originally the house vacuum was used to evacuate the furnace down to a pressure of about 10 mm. Hg. The house vacuum was then cut off and the final evacuation done with a diffusion pump. However, large variations in the blanks (from 0.0005 mg. to 0.05 mg.) led to investigating the house vacuum system as a source of CO_2. The house vacuum—as the name implies—is used by the whole laboratory, and consequently there is a large variation in the pressure, ranging from a few mm. to 2 or 3 cm. Hg when someone is evacuating a large volume. It is conceivable that a sudden increase in the line pressure may cause a reverse current into the analyzing system and if there is any CO_2 in the line this will be frozen out in the liquid air trap. It was consequently decided to do all evacuation with the backing pump for the diffusion pump, and this resulted in a decided improvement. The apparatus in its final shape is therefore as shown on Pages 88 and 93, using an individual pump for the evacuation.

With the furnaces cold and with no sample in the boat, but otherwise running the apparatus as usual, the amount of C obtained is only 0.002 mg. (with a variation of from 0.001 to 0.003 mg.).

With the furnaces heated as usual (to 600 and to 1000° C. respectively) and going through the regular procedure of analyzing a sample except that no sample is used, the following results are typical.

Table II

Blanks of Apparatus

Procedure same as usual, except no Sample in Boat.

Test No.	Tube Evac. Boat Cold		Boat heated in Vacuo to 600°			Boat heated to and at 1000° Tube filled with O_2, then evacuated.					
						1st Period			2nd Period		
	min.	Final Pressure mm.	min.	Final Pressure mm.	Carb. Obt'd mg.	min.	Final Pressure mm.	Carb. Obt'd mg.	min.	Final Pressure mm.	Carb. Obt'd mg.
104	23	0.010	11	0.000	0.013	27	0.010	0.029	..	.....	.....
108	14	0.015	20	0.003	0.027	26	0.010	0.025	25	0.010	0.030
124	51	0.002	20	0.001	0.017	36	0.005	0.038	40	0.005	0.044
Mean of 3 Tests					0.019			0.031			0.037
Variation from Mean					±0.007			±0.007			±0.007

The maximum variation in the blank is therefore less than ±0.010 mg. C, which for a 10 g. sample amounts to ±0.0001 per cent. For samples of different carbon contents the probable errors in the analysis are therefore as follows:

Table III

Probable Errors in Samples of Various Carbon Contents.

10 g. Samples.

Carbon Contents		Errors in Analysis	
Percent	Mg.	Mg. (±)	Percent (.)
0.1000	10	0.01	0.1
0.0100	1	0.01	1.0
0.0010	0.1	0.01	10.0
0.0001	0.01	0.01	100.0

Results

(a) *Electrolytic Iron.* A sample of doubly refined electrolytic iron serving as our standard of low carbon iron was analyzed according to the above method, giving the following results:

Table IV

Carbon Content of Electrolytically Refined Iron.

Standard Sample. 5 g. Samples.

Test Number	Sample Evac. Cold	Sample Heated in Vacuo to 600°*				Sample Heated to and at 1000°. Tube Filled with O_2 and Evac.								Net Carbon Obtained		
						1st Period				2d Period						
			Carbon Obtained				Carbon Obtained				Carbon Obtained			1st and 2d Car. Obt'd	Below 600° Adsorbed	At 1000° Combined	Total
	Min.	Min.	Gross Mg.	Blank Mg.	Net Mg.	Min	Gross Mg.	Blk. Mg.	Net Mg.	Min	Gross Mg.	Blk. Mg.	Net. Mg.	Net Mg.	Percent	Percent	Percent
110	18	20	0.286	0.019	0.267	31	0.219	0.031	0.188	29	0.093	0.037	0.056	0.244	0.0053	0.0049	0.0102
111	15	20	0.320	0.019	0.301	28	0.220	0.031	0.189	25	0.067	0.037	0.030	0.229	0.0060	0.0046	0.0106
112	15	20	0.295	0.019	0.276	28	0.235	0.031	0.204	29	0.076	0.037	0.039	0.243	0.0055	0.0049	0.0104
														Mean of Three Samples	0.0056	0.0048	0.0104
														Variation from Mean (±)	0.0004	0.0002	0.0002

* Carbon eliminated during 2d Period at 600° in Vacuo is < 0.01 mg.

These results show that the total carbon content of this material is 0.0104 (±0.0002) percent. The important fact to be noted from these results is that of the total carbon content, more than half (0.0056 (±0.0004) percent) is in the form of adsorbed gases, such as CO or CO_2, given off at or below 600°, and that the combined carbon content is only 0.0048 (±0.0002) percent.

The above standard sample of electrolytic iron was carefully analyzed by Mr. C. J. Rottmann, according to the "Old" method, the average of 11 separate analyses being 0.0177 percent with a maximum variation from the mean of ±0.0080 percent. This amount includes all carbonaceous matter introduced into the combustion tube, *i. e.*, CO_2 admitted in opening and closing the tube, adsorbed gases and combined carbon.

In order to determine the amount of CO_2 admitted to the combustion tube while removing the old sample and introducing the new one, the gas was analyzed during the preliminary evacuation with the sample cold. Six tests gave values of carbon introduced in this way varying from 0.15 mg. to 0.67 mg. Based on a 5 g. sample this corresponds to 0.0030—0.0134 percent. The results are tabulated in Table V for the sake of comparing the "Old" and the "New" methods.

Table V

Comparison of "Old" and "New" Methods of Analysis. Electrolytically Refined Iron. Standard Sample.

	"New" Method Mean of 3 Tests Percent C	"Old" Method Mean of 11 Tests Percent C
1. Carbon introduced while opening and closing combustion tube	0.0080±0.0050	
2. Carbon as adsorbed Gases	0.0056±0.0004	
3. Carbon in Combined Form	0.0048±0.0002	
Total,	0.0184±0.0056	0.0177±0.0080

The results are in remarkably close agreement, both as to total carbon and as to variation. It will be noted that the variation is largely due to item 1, namely carbonaceous gases introduced into the combustion tube while opening and closing the tube, gases that have no connection with the sample at all, but amounting to 30–130 percent of the total carbon content (items 2 and 3). This fact explains the large variation usually obtained in the carbon content of low-carbon iron.

(*b*) *Vacuum Fused Electrolytic Iron.* Table VI shows some results obtained with electrolitic iron after being melted in a vacuum furnace and forged into rods. 2–202 and 2–210 were melted in an Arsem type furnace and 2–251 in a Tungsten wound furnace.

(C) *Bureau of Standards Samples.* As a check of the constants of the apparatus two of the Bureau of Standards standard steel samples were analyzed, the results being given in Table VII.

Judging from these results the agreement is as close as can be expected, and the conclusion is justified that the constants of the apparatus are correct[5].

Modifications of the Method.

The apparatus as described in this paper is necessarily elaborate, especially the analyzing part of it, in order to eliminate all possible errors. However, great simplifications can be made to meet the requirement of different users. In another apparatus used in this laboratory the anlyzing system consists of two good stopcocks,

5 It should be noted that for such large carbon contents, the accuracy of the apparatus is not as great as for smaller carbon contents, because in the former case the pressures are too large to be read on the McLeod gage and must be read on the barometer directly. In the above cases the pressures were in the neighborhood of 20 mm., and the readings can be read with an accuracy of only ± 0.5 mm., resulting in probable errors of ± 2.5 percent.

Table VI

Vacuum Fused Electrolytic Iron. 5 g. *Samples.*

Test No.	Sample No.	Description of Sample	Net Carbon Obtained		
			600° Percent	1000° Percent	Total Percent
121	2-202	As forged. Outer half of Rod, except surface. Ether cleaned.....	0.0104	0.0159	0.0263
119	2-202	Forged and Annealed in Vacuo. All of Rod except surface. Not cleaned..	0.0000	0.0038	0.0038
120	2-202	Forged and Annealed in Vacuo. All of Rod except surface. Ether cleaned.	0.0000	0.0045	0.0045
115	2-210	As forged. Outer half of Rod, except surface. Not cleaned.....	0.0027	0.0042	0.0069
116	2-210	As forged. Outer half of Rod, except surface. Ether cleaned.....	0.0015	0.0039	0.0054
114	2-210	As forged. Center of Rod. Ether cleaned.....	0.0004	0.0030	0.0034
117	2-210	Forged and Annealed in Vacuo. All of Rod except surface. Not cleaned..	0.0009	0.0015	0.0024
122	2-251	As forged. Center of Rod. Ether cleaned.....	0.0006	0.0025	0.0031

Previous analyses by the "Old" method have given 0.02-0.04 percent Carbon for the above samples, *i. e.*, up to 10 times what the "New" method gives.

a liquid air trap, and a long mercury column similar to the arrangement shown on Page 88, and it gives very satisfactory results. Furthermore, split furnaces can be used, arranged on swinging arms, thus doing away with the long combustion tube. This modification would probably result in lower blanks, because the carbon obtained in the blanks is probably, in part at least, due to diffusion of CO_2 from the atmosphere through the heated portions of the tube. Finally the mercury diffusion pump may be eliminated, depending entirely on the oil pump for evacuation. This is done in the apparatus referred to above. In short, the apparatus can be made as simple or as elaborate as the requirements call for.

Summary and Conclusions.

1. It has been shown in this paper that by the method described great accuracy in determining the products of the combustion tube is possible; 0.001 mg. carbon can readily be measured by the analyzing system.

2. The blanks of the apparatus are small and consistent, the maximum variations amounting to only ± 0.007 mg. carbon, which is the probable error of the apparatus. This, for a 10 g. sample

containing 0.01 percent carbon would result in an error of ±0.00007 percent carbon or 0.7 percent of the total. This is 100 times better than is possible by any of the present methods.

Table VII

Analysis of Bureau of Standards Standard Samples.

2 g. Samples.

No. 20a Acid Open Hearth Steel, 0.4 percent Carbon. Containing, according to the Bureau of Standards, 0.393 percent Carbon.
No. 15a Basic Open Hearth Steel, 0.1 percent Carbon. Containing, according to the Bureau of Standards, 0.109 percent Carbon.

Test No.	Sample	Net Carbon Obtained		
		600° Percent	1000° Percent	Total Percent
102	No. 20a	0.015	0.367	0.381
103	No. 20a	0.010	0.365	0.375
	Mean	0.0125	0.366	0.378
	Variation from Mean (±)	0.0025	0.001	0.003
105	No. 15a	0.005	0.112	0.117
106	No. 15a	0.009	0.094	0.103
107	No. 15a	0.006	0.101	0.107
	Mean	0.007	0.102	0.109
	Variation from Mean (±)	0.002	0.010	0.007

3. By carrying out the analysis in three stages, namely (*a*) with the sample evacuated cold, (*b*) heated to 600° in vacuo, and (*c*) heated to 1000° in oxygen, it is shown that the total amount of carbon obtained from electrolytic iron is divided about equally between (*a*), (*b*) and (*c*); in other words, that about 0.005 percent is due to gases admitted to the tube in introducing the sample, that about 0.005 percent is present in the iron as adsorbed or occluded gas, and that the remainder 0.005 percent is the combined carbon.

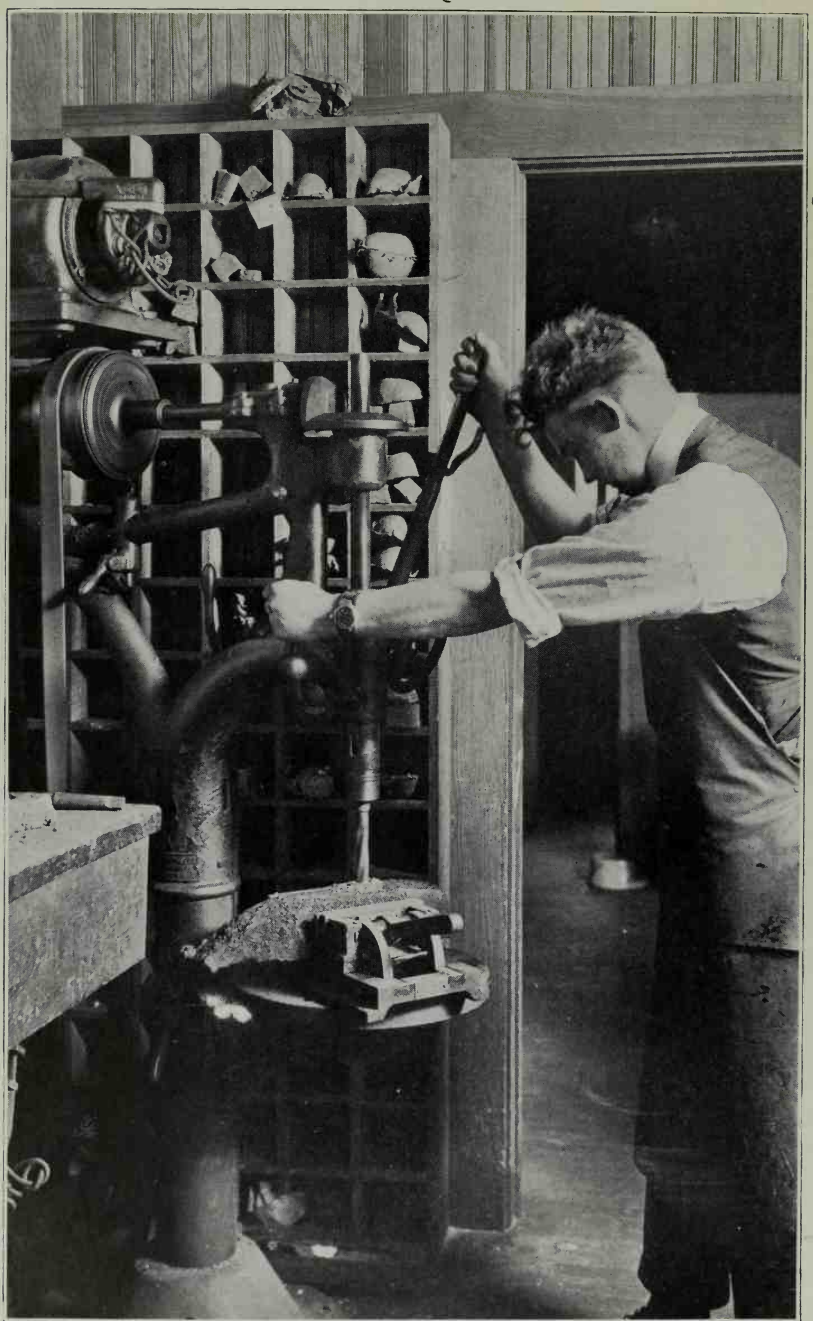
4. A great deal of variation exists in the amount of carbon obtained during the first stage of analysis, (3*a*), and it has been shown that the great variations in the carbon contents of low carbon iron usually obtained may be attributed to this source.

5. The new method can be modified to suit any given condition; but for carbon contents of 0.10 and above the established methods, when carefully carried out, are sufficiently reliable to make more refined methods unnecessary. It is only for very low carbon contents

that the errors in the established methods are so large as to conceal the true condition.

The author wishes to express his appreciation to Mr. A. L. Shields for his conscientious work in connection with the analyses and to Mr. C. J. Rottmann for his valuable advice based on his large experience in all matters pertaining to analytical chemistry.

Westinghouse Research Laboratory,
January 15, 1920.



Drilling pig iron for chemical analysis. All pig iron is carefully analyzed, five samples being taken from each car-load received

CARBON

AN ELECTROLYTIC RESISTANCE METHOD FOR DETERMINING CARBON IN STEEL[1]

By J. R. Cain and I. C. Maxwell[2]

INTRODUCTION

The purpose of this study was to investigate the accuracy, speed, practicability of a method for determining carbon in steel, dependent in principle on passing the carbon dioxide produced by direct combustion of the metal into a solution of barium hydroxide of known electrical resistance; after complete absorption of this gas the resistance is again determined and from the increase in this (due to precipitation of barium ions) the percentage of carbon is deduced. This method is new in principle and it is believed that the principle can be applied generally in many cases where the substance being determined precipitates another substance from solution with resultant change in resistance. The assembly of apparatus for determining resistance is also new,[3] and offers many advantages for technical work over the methods hitherto in general use for measurement of electrolytic resistances, which require the use of induction coils or high frequency generators, tuned telephones, balanced inductances and capacities, etc. Other new features are the application of the nomograph[4] for the graphical representation of resistance data and the use of special conductivity cells with adjustable electrodes to facilitate the manufacture of any number of such cells with the same cell constant.

Much work has been done by others on electro-chemical analytical methods. In general, these fall into three groups in which an end-

1 Complete equipment for determination of carbon by this method may be obtained from Arthur H. Thomas Co., Philadelphia.

2 Journal of Industrial and Engineering Chemistry, Vol. 11, No. 9, page 852. September, 1919.

3 The elements of this were described by Weibel and Thuras, This Journal, **10** (1918), 626.

4 The mathematical work in constructing the nomograph shown on page 112 was done by Mr. H. M. Roeser of the Bureau of Standards at the request of the senior author, who suggested its application to electrolytic resistance data. A paper on this subject is in preparation by Mr. Roeser. References on the nomograph are: "Traite de Nomographie," by M. d'Ocagne, Gauthiers-Villars, Paris; "Graphical Methods," by Carl Runge, Columbia University Press, New York; "Graphical Interpolation of Tabulated Data," by H. G. Deming, *J. Am. Chem. Soc.*, **39** (1917), 2388; "The Nomon, a Calculating Device for Chemists," by H. G. Deming, *Ibid.*, **39** (1917), 2137.

point is shown electrochemically by the following methods: (1) The unknown concentration is obtained from curves expressing a relation between cubic centimeters of titrating solution and conductivity (or a related quantity) of the solution titrated;[1] (2) the unknown is obtained from curves giving the relation between cubic centimeters of titrating solution added and the corresponding electromotive forces of a cell composed of a normal electrode and an electrode not acted upon by the solution being titrated, the latter being the electrolyte;[2] (3) special application of Method 2 used for determining hydrogen ion in acidimetry and alkalimetry and in precipitations from neutralized solutions.[3]

Such methods suffer by comparison with the present for the following reasons: (1) A curve has to be plotted for every determination, which consumes much time; (2) the apparatus required to determine carbon with an accuracy of 0.01 per cent carbon would be too delicate and inconvenient of manipulation for every-day use; (3) the difficulty in some cases of fixing with sufficient definiteness the inflection or break in the curve denoting the end-point of the titration. Upon further comparing these methods with the present, it is seen that the latter dispenses with one operation common to all the others, namely, the addition of successive portions of a titrating solution and the determination of the resistance at each addition, resulting in additional time-saving.

From an inspection of the chemical equation for the reaction underlying the present method,

$$Ba(OH)_2 + CO_2 = BaCO_3 + H_2O,$$

it is evident (when any given conductivity cell is used) that the only factors which act to change the conductivity of the barium hydroxide used for absorption are (1) the amount of carbon dioxide absorbed, which determines the disappearance from solution of the barium ion, and (2) the temperature. Since carbon dioxide precipitates barium without leaving reaction products in the solution to increase the conductivity (such as would remain if, for instance, sodium sulfate were the precipitating agent for the barium) it can be seen that the present

1 Harned, *J. Am. Chem. Soc.*, **39** (1916), 252; Findlay, "Practical Physical Chemistry," Ostwald-Luther, "Physikalisch-Chemische Messungen."

2 Loomis and Acree, *Am. Chem. J.*, **46** (1911), 585, 621 (a bibliography is also given); Hildebrand, *J. Am., Chem. Soc.*, **35** (1913), 869; Kelly, *Ibid.*, **38** (1916,) 341.

3 Hildebrand, *Loc. cit.* See also Weibel and Thuras, Journal of Industrial and Engineering Chemistry, **10** (1918), 626, for another electrolytic method.

method should give the maximum possible change of resistance for a given amount of barium removed—a condition tending to secure a high degree of sensitiveness.[1] However, the temperature coefficients of resistance of barium hydroxide solutions in the range of concentrations herein employed average nearly 1.7 per cent per degree, hence it is evident that the accuracy of the method will be largely affected by temperature if due correction is not made.

In developing this method it was deemed necessary: (1) To construct the curve showing resistance as a function of concentration of barium hydroxide solutions ranging from very concentrated to very dilute and to select the portion of this curve showing the maximum change of resistance for a given change of concentration; (2) to devise an apparatus which, when the selected barium hydroxide solutions were used in it, would completely absorb the carbon dioxide at the highest rates of passage of the gas current. The same absorption apparatus, in order that the method might meet the requirements of convenience and rapidity, should permit resistance determinations to be made without transfer of the solution to another vessel; it should also be easy to fill and empty; (3) to determine the temperature coefficients of the barium hydroxide solutions in the selected range of concentration;[2] (4) to prepare a chart enabling the operator to read directly therefrom the percentage of carbon, all temperature corrections being incorporated; (5) to design an apparatus for the electrical measurements possessing the necessary robustness, reliability, simplicity, and protection from corrosion by the laboratory fumes.

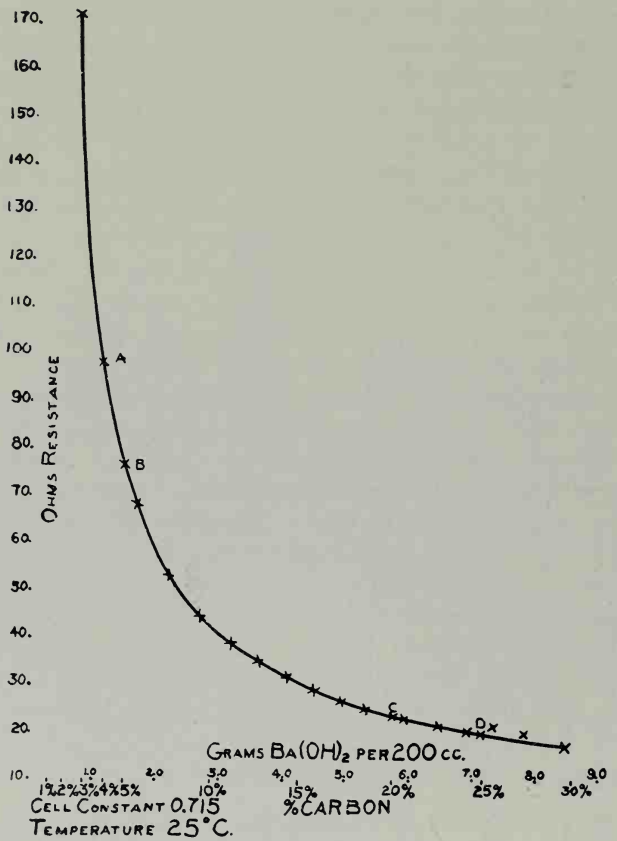
The Resistance of Barium Hydroxide Solutions

A curve was prepared showing the relation between electrical resistance and barium hydroxide concentration when the latter was varied from practically saturation to nearly zero. The data in the literature being insufficient for this purpose, the determinations were made by Mr. Louis Jordan of this Bureau and are represented on Page 104. The solutions for constructing the curve were prepared from J. T. Baker's analyzed barium hydroxide by diluting a stock solution of this with carbon dioxide-free water and determining their strength by titration against standard hydrochloric acid using methyl orange as indicator. The resistance measurements were all made in the same conductivity cell and at practically the same

1 Compare the conditions in Harned's work with $Ba(OH)_2$ solutions. *Loc. cit.*

2 There is some possibility of placing the absorption vessel in a constant temperature bath together with a compensating cell in the other arm of the bridge. This would remove the necessity for temperature correction. The method described herein, however, is believed to be simpler.

temperature (27° C.)[1]. No great accuracy is claimed for these results, which are used only for establishing the form of the curve and



selecting a portion of it for more exact redetermination. The portion of the curve selected for use in this method is that between A and B. This region gives the maximum change of resistance per unit change of concentration consistent with the use of solutions sufficiently concentrated to effect complete absorptions of carbon dioxide under the conditions imposed. Comparing the resistance changes with changes of concentration of barium hydroxide corresponding to 1 per cent carbon on different parts of the curve, it is seen, for example, that these changes in resistance are approximately six times as great on the portion

[1] They were not corrected by any temperature coefficient since, as an inspection of Table 1 shows, they were made at near enough to one temperature to give roughly the form of the curve, which was all that was desired.

AB as on the portion CD. The use of the most dilute solutions possible is, of course, also desirable from the saving in barium hydroxide effected. Solutions to the left of A, even when used in very efficient absorbing vessels will not retain all the carbon dioxide except at rather slow rates of aspiration.

TABLE I—Data for Resistance-ConcentrationCurve for Ba(OH) Solutions in the Region 2 Per Cent to 30 Per Cent Carbon Equivalent Strength

Cell Constant = 0.715

$Ba(OH)_2$ per 200 cc. Soln. Grams	Equivalent[1] Per cent Carbon	Temperature Deg C.	Resistance Ohms
0.612	2.14	27.9	171.6
1.12	3.92	27.3	97.9
1.68	5.87	27.3	68.5
2.20	7.70	27.3	53.5
2.67	9.35	27.3	44.9
3.17	11.1	27.2	38.7
3.64	12.7	27.0	34.6
4.05	14.2	26.9	31.2
4.43	13.5	27.0	29.2
4.94	17.9	27.2	26.2
5.31	18.6	27.0	24.7
5.88	20.6	26.9	22.7
6.45	22.5	26.0	21.8
6.93	24.2	26.8	20.1
7.34	25.6	21.2	20.8
7.84	27.4	21.2	19.7
8.45	29.5	27.3	16.4

1 The method used in Tables I and II and elsewhere throughout this paper for expressing the strength of the barium solution in terms of "equivalent per cent carbon" was chosen for convenience in using the nomograph described subsequently. By "equivalent per cent of carbon" is meant the amount of carbon expressed as percentage on the basis of 2-g. samples being used, necessary to precipitate completely all the barium ions from the solution concerned. This amount of carbon is calculated from the equation; $Ba(OH)_2 + CO_2 = BaCO_3 + H_2O$. For instance, in the first horizontal column of this table it is seen that 2.14 "equivalent per cent carbon" corresponds to 0.612 g. $Ba(OH)_2$; the former figure was obtained by solution of the proportion:

Mol. Wt. $Ba(OH)_2$: At. Wt. Carbon :: Wt. of $Ba(OH)_2$ in 200 cc. Soln. : Wt. of Carbon in Sample

or

171.38 : 12.00 :: 0.612 : X

whence X = 0.0428 g. carbon

or the "equivalent per cent carbon" = $(x/2)100 = 2.14$.

The portion of the curve selected for use by this method is again shown on Page 107. The procedure used in determining the points on it was the same as for constructing the curve shown on Page 104, except that more care was taken to secure accurate readings and the results were corrected by the coefficients given under the heading "Temperature Coefficients." Table II gives the data used in constructing this curve.

TABLE II—Data for Resistance-Concentration Curve of $Ba(OH)_2$ Solutions in the Region 4 Per cent to 5 Per cent Carbon Equivalent Strength
Cell Constant = 0.715

$Ba(OH)_2$ per 200 cc. Soln. Grams	Equivalent Per cent Carbon	Temperature Deg. C.	Observed Resistance Ohms	Corrected Resistance Ohms
1.164	4.075	23.6	100.9	98.49
1.198	4.194	24.2	97.4	96.08
1.239	4.337	24.2	94.6	93.26
1.242	4.348	23.2	95.8	92.81
1.280	4.482	24.4	91.4	90.42
1.286	4.500	26.6	87.6	90.01
1.301	4.553	27.5	85.3	88.92
1.303	4.559	26.4	86.8	88.79
1.346	4.712	25.1	86.0	86.06
1.385	4.848	25.8	82.7	83.90
1.403	4.912	24.4	84.2	83.94
1.460	5.110	23.1	82.8	80.11

The Absorption Apparatus

The essential features desirable in the absorption apparatus are: (1) It must retain all carbon dioxide when gas passes at the rate of 300 to 400 cc. per min.; (2) it must permit resistance measurements to be made without transfer of the solution to another cell; (3) temperatures of solutions should be easily read to 0.1°; (4) the cell should be easy to clean and fill with fresh solution; (5) all cells should be built with the same cell constant,[1] or the latter should be capable of adjustment to one value for all, so that the chart or set of tables may be used for all cells.

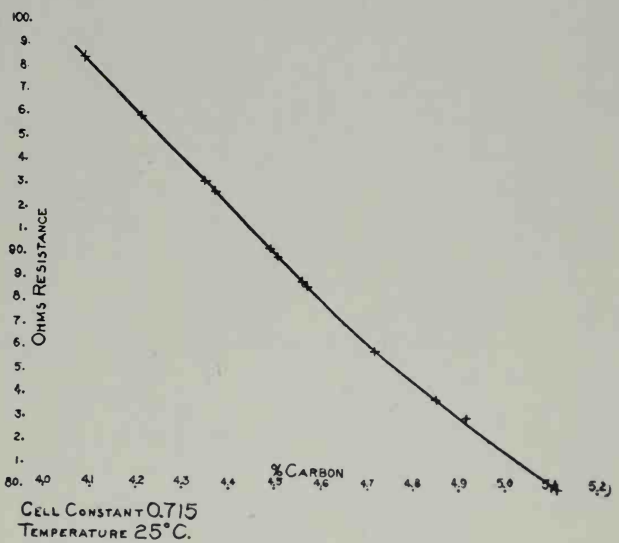
Fleming[2] and others have shown that the combustion of a sample of steel and the sweeping out of the products of combustion from the apparatus where combustion tubes of the usual length and bore are used, can be accomplished in 5 to 6 min.; this requires passage of the oxygen at the rate of about 300 to 400 cc. per min. Soda lime has been found to absorb all the carbon dioxide at this and higher rates. However, barium hydroxide solutions of all concentrations are much less efficient absorbents than soda lime. The absorbing efficiency increases slightly with the concentration, but even when the most concentrated solutions were used it was impossible to absorb completely all the carbon dioxide in the usual types of gas absorption vessels, nearly all of which were tried. The method of testing these forms of apparatus was to burn a sample of steel, having a gas meter before the furnace to control the rate of passage of the oxygen, and to

1 The "cell constant" is not a constant at all, as Washbourne and others have shown, but for want of a better term this expression has been used throughout this paper.

2 *Iron Age*, **93** (1913), 64.

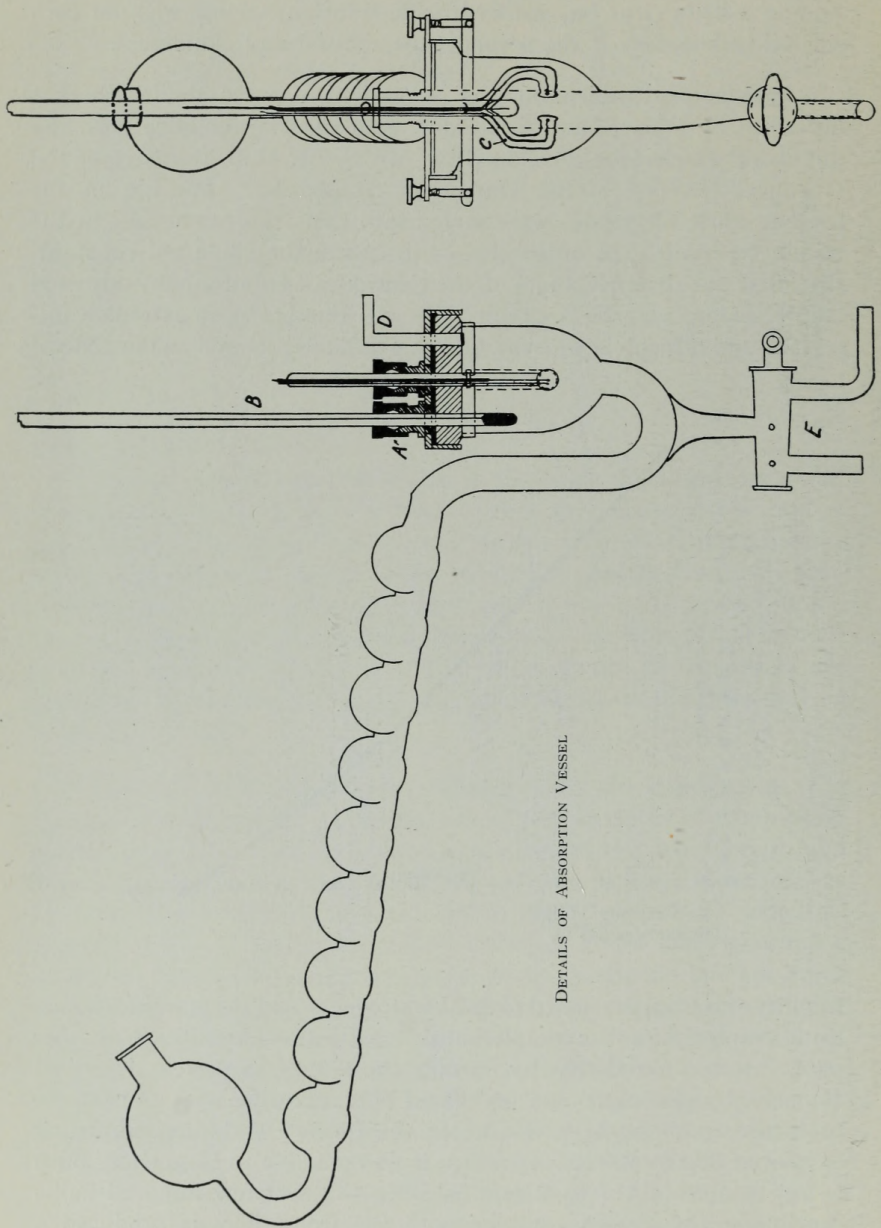
attach after the tube being tested a second absorption vessel containing a little clear barium hydroxide solution; a test was not considered satisfactory if the second tube showed any cloudiness.

Satisfactory absorption was secured in a vessel similar to that described by Weaver and Edwards,[1] with suitable modifications, the details of which were developed by Mr. S. M. Hull, formerly of the Chemical Division of the Bureau of Standards. For use in the present work, electrodes were sealed into this absorption tube in the middle reservoir. In order always to secure the same cell constant, the area and distance apart of the electrodes were originally adjusted before sealing into the reservoir. This was found to be an extremely uncertain and difficult operation, and this difficulty as well as the general



fragility of the apparatus led to its abandonment and the search for something simpler. After the completion of investigations described in another paper,[2] it was found that by burning the sample as therein described (namely, by placing it on a preheated boat, allowing boat and sample to further preheat for a minute in the furnace and then admitting oxygen at 300 to 400 cc. per min.), it was possible to completely burn a 2-g. sample in 1½ to 2 min. Since the first few hundred cubic centimeters of oxygen combine with the iron, there is produced a

1 Journal of Industrial and Engineering Chemistry, **7** (1915), 534.
2 Cain and Maxwell, Journal of Industrial and Engineering Chemistry, **10** (1918), 520.



DETAILS OF ABSORPTION VESSEL

much better partial pressure of carbon dioxide than is secured where a sample burns slowly and this makes it possible to use a very simple absorption tube, such as that shown on Page 108, for the carbon dioxide.

It was also found simpler to build a cell whose electrodes could be adjusted to secure a given cell constant than to attempt to obtain the same result with the electrodes sealed in a fixed position in the cell. This fact and the use of the special method of combustion described led to the design of the apparatus shown on Page 108. This is not fragile and the glass parts can be built by any glass blower of ordinary skill; it meets all the listed requirements of the absorption apparatus.

The adjustment of the cell constant is made by moving the electrodes up and down after loosening the stuffing box; a marked change takes place as they approach the meniscus. Initial approximate similarity can be attained by the glass blower with comparative ease. Once the electrodes are set in the proper position, this is maintained by cementing with DeKhotinsky cement. The electrodes are platinized and the cell constant is determined as described under "Operating Suggestions." A certain amount of adjustment of the constant of the cell can be secured by removing or adding platinum black to one or the other of the electrodes during the platinizing operation by the use of an auxiliary electrode. Some cells in long use at the Bureau have differed in cell constant by 0.04 without affecting the accuracy of results. Actually, it is easily possible to adjust cell constants within 0.005, and this practice is to be recommended.

Temperature Coefficients

The solutions for determination of these coefficients were prepared and standardized as already described. The conductivity cells used for the work were kept in a thermostatically controlled chamber where the temperatures were maintained within 0.01° C.

Resistances of barium hydroxide solutions equivalent to approximately 4.0, 4.25, 4.50, 4.75, and 5.0 per cent carbon were determined at 20°, 25°, and 30°. The experimental values and the corresponding calculated values for a and B are shown in Table III. These values for a and B were calculated by substituting the values for temperature and resistance from Table III for t in the equation

$$\frac{1}{R_t} = \frac{1 + a[t-25] + B[t-25]^2}{R_{25}},$$

and solving for a and B.

Methods for Direct Reading of Carbon Percentages

Several methods were tried for graphically representing the relation between carbon percentages and the corresponding observed temperature and resistance measurements. At first tables were constructed in which temperature corrections were calculated and applied for every tenth of a degree and every tenth of an ohm, but these were found to be too cumbersome. Other tables were then constructed giving the temperature corrections only, with the idea of adding or subtracting these each time a reading was made. This condensed the tables very considerably but increased the amount of calculation necessary. A series of curves was then constructed showing the relation between resistance and per cent carbon, a curve being constructed for every tenth of a degree. Such curves are convenient to use only if they are plotted on an inconveniently large scale; even then it is very fatiguing to use them for many readings at a time.

TABLE III—Data for Temperature Coefficients of Resistance

Concn. of $Ba(OH)_2$ Soln.[1] Per cent C. Approx.	Temperature Deg. C.	Resistance Ohms	a	$B \times 10^{-4}$
5.0	20	135.25	0.01674	0.2687
	25	124.02		
	30	114.37		
4.75	20	142.18		
	25	130.36	0.01680	0.3505
	30	120.16		
4.50	20	149.07		
	25	136.62	0.01686	0.3085
	30	125.91		
4.25	20	156.18		
	25	143.07	0.01687	0.1652
	30	131.89		
4.00	20	165.18		
	25	151.28	0.01687	0.0898
	30	139.48		

1—200 cc. of the solution contained barium hydroxide approximately equivalent to the carbon in a 2-g. sample with 5 per cent C, or 100 cc. of the solution contained barium hydroxide approximately equivalent to the carbon in a 1-g. sample with 5 per cent C; or approximately 0.7125 g. $Ba(OH)_2$. The other solutions contained barium hydroxide approximately equivalent to 1-g. samples with 4.75 per cent, 4.50 per cent, and 4.25 per cent, 4.00 per cent, respectively.

After a little study it was found that the B term could be omitted from the equation for correcting temperatures to 25°, provided this equation is applied only between the temperatures 15° and 35°; at higher or lower temperatures the correction for the B term is appreciable. If the laboratory temperature is not within these limits,

the stock solution in carboy F on Page 114 must be brought within this range by placing it in a bath of cold water. The equation

$$(1)\ R_{25} = \frac{Rt[1 + a(t - 25) + B(t - 25)^2}{Rt}$$ then reduces to

$R_{25} = Rt[1 + a(t - 25)]$, in which $a = 0.01681$, this value being taken from Table III. After trials of other forms of curves, it was found that the parabolic form answered the requirements of fitting the observations sufficiently well and of permitting the construction of a nomograph; the equation to the curve shown on Page 107 was calculated on the assumption that it was parabolic, using the methods of least squares and the observations shown in Table II. This equation was found to be

$$C^2 - 13.589\ C + 63.191 - 0.2478\ R_{25} = 0$$

or,

$C^2 - 13.589\ C + 63.191 - 0.2478\ [1 + a(t - 25)] = 0$ whence,

(2) $C = 6.79 - \frac{1}{2}\sqrt{0.9912R(0.5798 + .01681t - 67.11)}$, C being the equivalent per cent of carbon in the sense already explained. From Equation 2 the nomograph shown on Page 112 was prepared. This nomograph can be used for all cells having cell constants not too different from 0.715, which was the cell constant of the cell used when data for the curve shown on Page 107 were obtained. As has been shown, the form of cell used allows the electrodes to be adjusted always to secure this result. (Page 108.)

The use of the nomograph is very simple. A straight edge is made to correct the observed values for temperature and resistance and the intersection with the third (middle) ordinate gives the concentration of the solution in terms of carbon percentage; this may be termed the first concentration. After the combustion is ended a similar set of readings is taken and subtraction of the second concentration reading from the first gives directly the per cent of carbon if a 2-g. sample has been taken; or, if a 1-g. sample has been used, the result is multiplied by two. The scales can be read to 0.005 per cent C, 0.05° C., and 0.05 ohm. It was found by comparison of chart readings in a number of cases with the resistances computed by Equation 1 that the error of the chart is less than 0.005 per cent carbon.

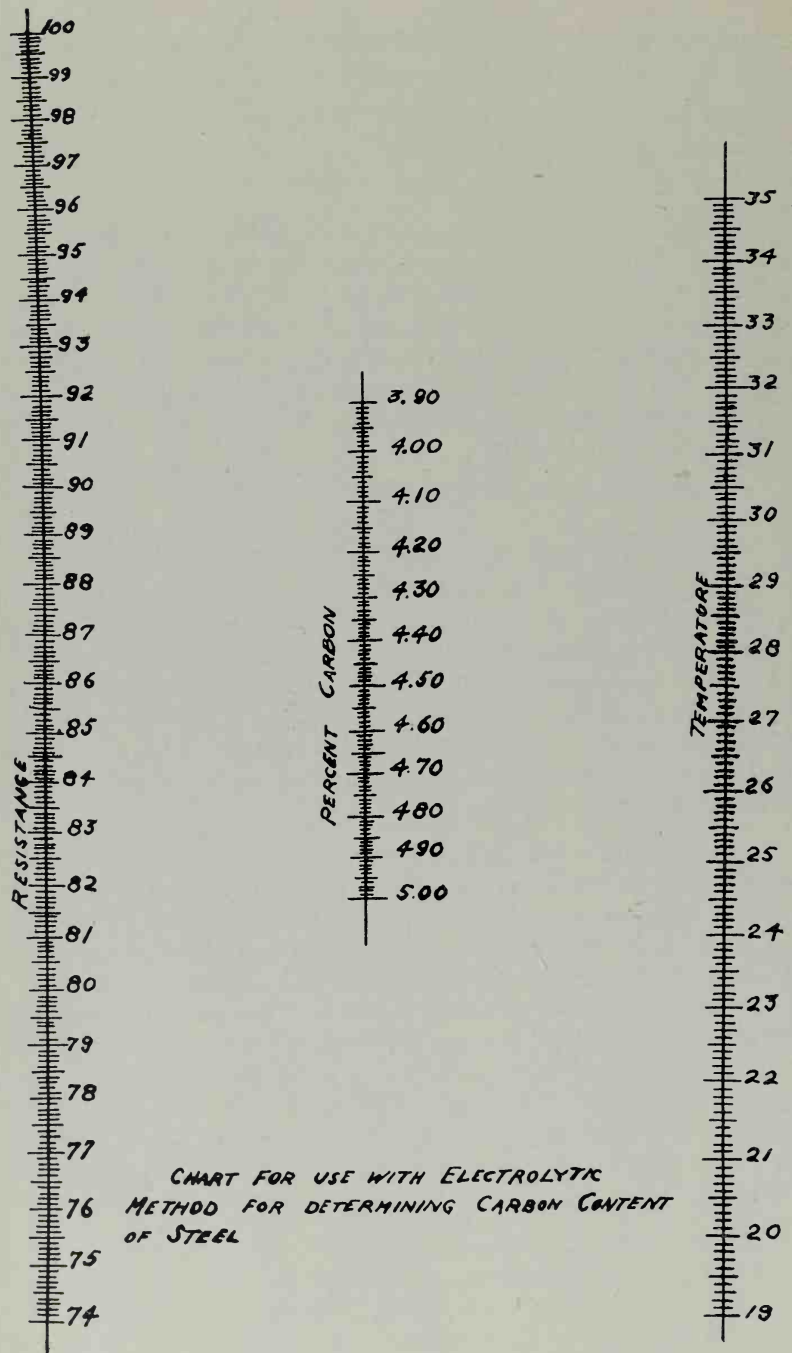


CHART FOR USE WITH ELECTROLYTIC METHOD FOR DETERMINING CARBON CONTENT OF STEEL

Apparatus for Determining Electrical Resistance

With the co-operation of the Leeds and Northrup Company, different forms of apparatus for measuring electrolytic resistances were built and tried. Two forms of high frequency generator in connection with tuned telephones were used; also one or two forms of induction coil as sources of alternating current, and an interrupter such as is used in wireless investigations; the latter was quite unsatisfactory, as might be expected, due to polarization. Good results were obtained with the high frequency generators and tuned telephones, provided that inductances and capacities were used as directed by recent investigators. Such refinements are inconvenient in a method such as the present, intended for works use, and there are two other important objections from the same standpoint: (1) the difficulty of detecting a minimum in the telephone when working in a noisy place such as the usual works laboratory and the fatigue of the operator who would have to make possibly 80 or 90 determinations in an 8-hr. day, and (2) the liability to deterioration of a high frequency generator when operating continuously 24 hrs. per day as might happen in technical use. These considerations led to the development of a method of measuring electrolytic resistances by the use of commercial 60- or 25-cycle current. It was found after trial of the Weibel[1] galvanometer, a vibration galvanometer and of a direct current galvanometer operating off a crystal rectifier placed in the secondary of a transformer (the cell being in the primary), that the Weibel galvanometer offered a very satisfactory zero instrument for such measurements of electrolytic resistance as had to be made in this work.

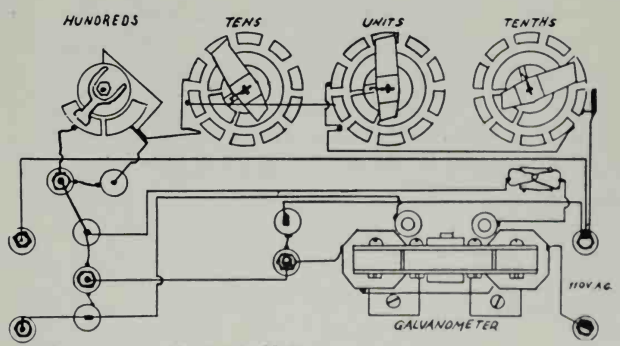
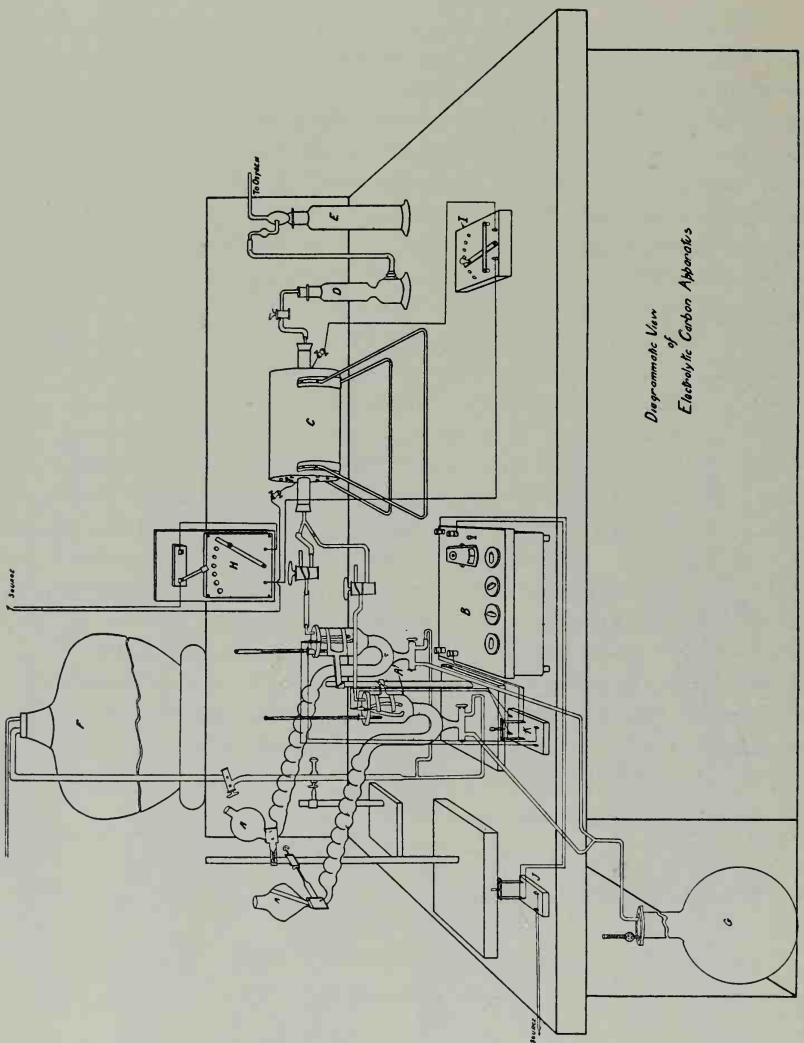


Diagram of Bridge Used for Resistance Measurements

The bridge shown on Page 113 was accordingly constructed by Leeds and Northrup. The resistance coils and galvanometer are enclosed in a hermetically sealed box so that they are efficiently protected from cor-

1 E. E. Weibel, Bureau of Standards, *Scientific Paper*, **297** (1917).



rosion. There are three dials—tens, units, and tenth ohms, respectively—and a fourth which adds 100 to the readings of the others, when this is desired. An accuracy of 0.1 ohm in the readings is all that is necessary, although much better than this can be done. This instrument has been in use several months and has been very satisfactory. A perfect minimum is always registered by the galvanometer, provided the electrodes are well platinized.

Procedure for Determining Carbon

A nichrome-wound furnace is used for heating. Porcelain or glazed quartz tubes may be employed; they should be fitted with a sheet nickel sleeve to protect internally from fused iron oxide which is thrown off from the burning steel. The two absorption vessels are filled to the 200 cc. mark with barium hydroxide solution from the stock bottle F (Page 114; see also Operating Suggestion 1). Oxygen or air freed from carbon dioxide is passed for a few seconds to mix the solutions and their temperatures and resistances are then recorded. In the meantime the combustion boat filled with alundum sand has been preheating in the furnace, which for this work should be maintained continuously at 1050° to 1100°, perferably the latter temperature.[1] This is an extremely important point, for if the temperature is too low or the oxygen is not pure or is not admitted at 300 to 400 cc. per min. after the combustion starts, the rapid combustion essential to successful absorption cannot be secured. The boat is removed from the furnace and when at a low red heat the sample is distributed on the alundum[2] and the boat replaced in the furnace and left to preheat, without oxygen passing, while the next sample is being weighed. Oxygen is now passed at the rate of 300 to 400 cc. per min. for the next two minutes; then the stopcock, is turned to the position, which admits carbon dioxide-free air; this should pass at the same rate as the oxygen. During this combustion period, if directions have been followed, all carbon dioxide will have been removed from the furnace, but some still remains in the large bulb of the absorption apparatus. The air removes this. The advantage of the use of air at this stage is obvious: a saving of oxygen is effected and the furnace is immediately available for burning the next sample. While air is passing through the first tube (requiring 1½ to 2 min.) the combustion of the next sample proceeds as already directed, using the second absorption tube. The second reading of resistance and temperature on the first tube then follows, and if the solution is not too dilute it can be used for absorbing the carbon dioxide from other samples; otherwise, a little is allowed to flow into the reservoir G, and the tube is filled to the mark again with fresh solution. Of course it is an economy in time for the operator, wherever possible, to choose conditions (weight of sample, carbon content of same, etc.) so as to get the maximum number of determinations for a single filling, since in this way the

1 The melting point of gold is a convenient temperature check. If this metal is not melted the temperature is too low. See paper cited, by Cain and Maxwell.

2 Experience has shown that no loss of carbon occurs unless the sample contains extremely fine particles; with most steels these can first be removed without causing an error in the carbon determination. This point should always be tested, however, in burning new steels.

second resistance and temperature readings serve as initial values for the next combustion and so on. The solution should not be used when it is more dilute than corresponds to 4 per cent carbon (*i. e.*, 99.5 ohms at 25° ; see nomograph, on Page 112, for the limiting resistances corresponding to other temperatures), since its absorptive power at rapid rates of passage of the oxygen is then less, and some carbon dioxide may be lost. The data relating to combustions should be recorded as obtained. It is convenient to use a tabular form for record showing: (1) Designation of sample; (2) weight taken; (3) cell used; (4) initial temperature; (5) final temperature; (6) initial resistance; (7) final resistance; (8) initial concentration; (9) final concentration; (10) carbon percentage in sample; (11) remarks. There is ample time for entering this information while other operations are going on. A very good way is to enter "final temperature" below "initial temperature," and "final resistance" after "initial resistance" for each sample, since this relates these quantities in an easy manner for reading the nomograph.

The speed of the method naturally depends on the skill of the operator and the ingenuity displayed in arranging a cycle of operations which secures the best speed under his working conditions. Operators at the Bureau of Standards when working on a series of Bureau of Standards' analyzed samples averaged one determination per 4½ to 5 min. The accuracy of results is shown in Table IV.

TABLE IV—Results by Electrolytic Resistance Method

B. S. Standard Sample Used	Wt. Used Grams	B. S. Value for Carbon (Av. by Direct Combustion) Per cent	Value by Electrolytic Resistance Method	Analyst
11*a*	2.0	0.223	0.21	Maxwell
11*a*	2.0		0.21	
12*b*	2.0	0.409	0.41	
	2.0		0.41	Swindells
	1.0		0.42	
14*a*	2.0	0.813	0.81	
	1.0		0.82	Swindells
	2.0		0.80	Maxwell
16*a*	0.5	0.990	1.00	
	0.5		1.00	
	1.0		1.00	Cain
	2.0		0.98	
21*a*	2.0	0.617	0.62	Cain
	2.0		0.62	Maxwell
	Gram		Gram	
Sugar	0.00421		0.0046	Maxwell
	0.00421		0.0042	Maxwell
	0.00421		0.0040	Maxwell

Operating Suggestions

1—Stock barium hydroxide solutions are conveniently made in one to two carboy lots by adding solid barium hydroxide to the carboy nearly filled with water (agitating with air) until the equivalent strength approaches 5 per cent carbon; subsequent additions can then be made by adding a saturated barium hydroxide solution. Of course, it is not necessary to make up exactly to the equivalent of 5 per cent carbon; an approximation to this is all that is desired. The strength of the solution is determined from time to time during the standardization by running a portion of the solution into the cell and measuring its resistance. If a set-up like that shown on Page 114 is used in measuring the resistance, it is not necessary to remove the carboy from the shelf or to break any of the connections during these operations. If it is desired to economize in the use of barium salt, the waste solution in reservoir G can be brought up to strength as described, after first decanting it off from the barium carbonate that has settled out. A still further economy can be effected by drying and calcining to oxide the precipitated barium carbonate; this oxide can then, of course, be used again.

2—The cell constants should be checked from time to time. This may be done (1) by turning standard samples, or (2) by determining the resistance of a $N/10$ solution of pure potassium chloride. This solution should be prepared on the day it is used, since it has been found that stock solutions change during the course of this work. Table V shows the resistivities at various temperatures of $N/10$ potassium chloride solutions. The cell constant is computed from the formula $R = NS$, or $N = R/S$, where R is the observed resistance, N the cell constant, and S the resistivity, taken from Table V, for the same temperature. If a change in cell constant has taken place it is most probable that the electrodes need replatinizing. Directions for this are given below. If Method 1 is used, any marked deviation from the correct carbon value of the standard steel may be due to a change in the cell constant, and this should then be checked by Method 2, unless the error in the carbon determination is suspected to be due to some other cause. In the present work no deviation of cell constants has been observed until after several months' use, and then the change is sudden and erratic. When the cell constant changes it should be brought back to the original value by the methods already described under the heading "The Absorption Apparatus."

3—If the absorption vessels are not to be used for some time they should be cleaned with hydrochloric acid (not over 2 to 3 per cent

HCl) followed by distilled water. Extreme care should be taken that none of the hydrochloric acid or chlorides enters the reservoir for waste barium hydroxide solution, if this is to be used again.

4—To platinize the electrodes, the cap carrying them is removed from the cell and they are first cleaned with sulfuric acid and chromic acid mixture followed by distilled water. Then they are immersed in a vertical position in a solution made of 100 g. water, 3 g. chloroplatinic acid, and 0.02 to 0.03 g. lead acetate. Current is passed through the solution by connecting the electrodes to three dry batteries in series; the current is passed for 5 min., reversing

TABLE V—Specific Resistivity of *N*/10 KCl Solution at Various Temperatures (from Landolt-Börnstein Physickalish-Chemische Tabellen, 4th Ed., Page 1117).

Temperature Deg. C.	Resistance Ohms
15.0	95.42
16.0	93.28
17.0	91.32
18.0	89.37
19.0	87.49
20.0	85.69
21.0	83.96
22.0	82.30
23.0	80.71
24.0	79.11
25.0	77.64
26.0	76.16
27.0	74.79
28.0	73.42
29.0	72.10
30.0	70.82
31.0	69.59
32.0	68.40
33.0	67.20
34.0	66.09
35.0	64.98

every half minute. Finally, an auxiliary platinum electrode is introduced and current passed with this as anode for another 2 min., after which the electrodes are washed thoroughly with distilled water and are then ready for use.

Summary

I—This paper describes the fundamental principles of a method for determining carbon dioxide by absorbing it in barium hydroxide solution and measuring the resistance change of the solution in relation to its concentration.

II—A suitable absorption vessel with electrolytic resistance cell incorporated is illustrated and described.

III—Resistance measurements of barium hydroxide solutions varying in connection from 3.08 g. $Ba(OH)_2$ per l. to 42.25 g. $Ba(OH)_2$ per l. were determined approximately, and determinations accurate within 0.01 ohm were made of the resistances of twelve different solutions varying from 5.820 g. $Ba(OH)_2$ per l. to 7.300 g. $Ba(OH)_2$ $(OH)_2$ per l.

IV—Temperature coefficients of resistance of these twelve solutions were determined in the range 20.00° to 30.00° C.

V—Based on the measurements of resistance of the barium hydroxide solutions, solutions with concentrations varying between 5.820 g. $Ba(OH)_2$ per l. and 7.300 g. $Ba(OH)_2$ per l. were chosen as most suitable for this method.

VI—Special resistance-measuring apparatus was developed which simplified these measurements by dispensing with tuned telephones, high frequency generators, and balanced inductances and capacities.

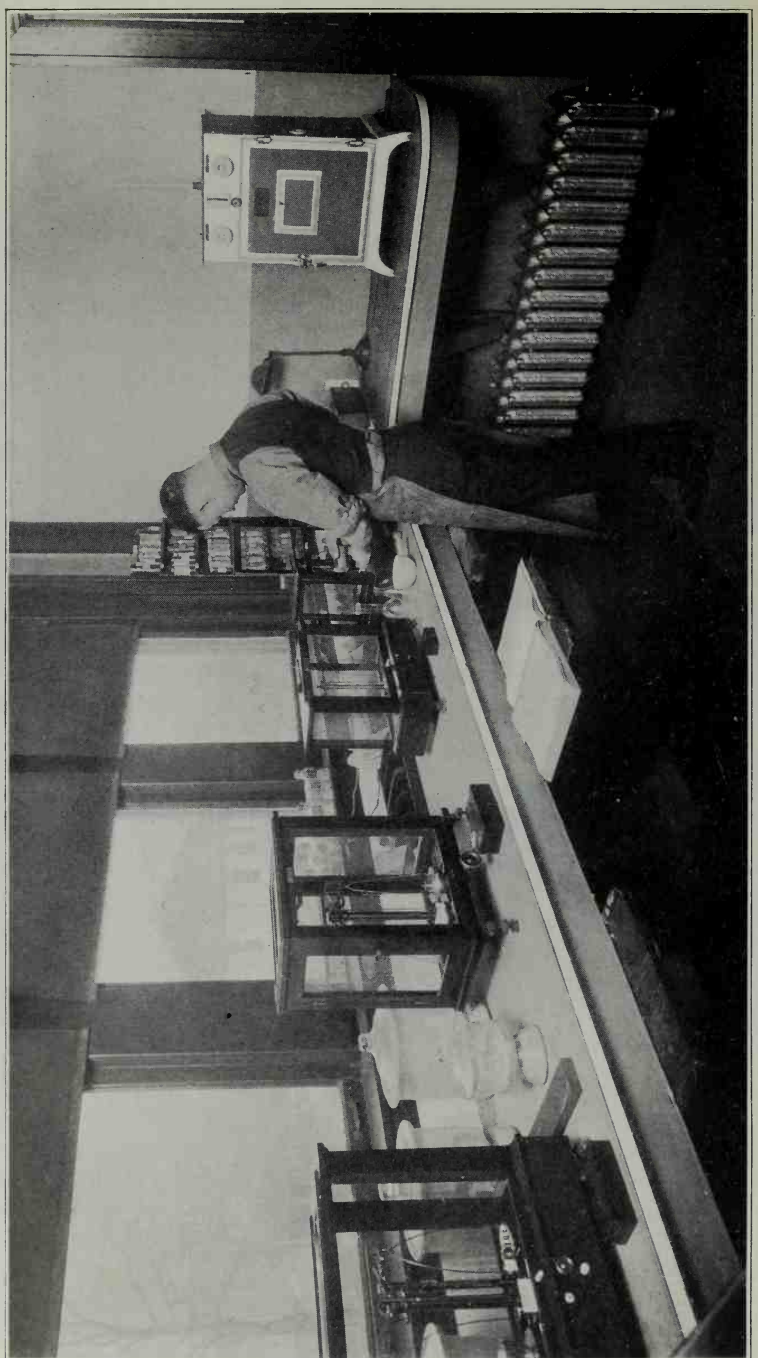
VII—A convenient nomographic method of applying necessary temperature corrections to resistance measurements was developed.

VIII—The method permits of accurate determinations of carbon in steel (*i. e.*, within 0.01 per cent carbon), by the direct combustion method in 4½ to 5 min.

Acknowledgment

The authors desire to acknowledge the work of Mr. H. E. Cleaves, former member of the Chemistry Division of the Bureau of Standards, who carried out some preliminary measurements, and the assistance of Messrs. Silsbee, Agnew, and Isler of the Electrical Division of the Bureau, who co-operated effectively in the selection of a suitable alternating current galvanometer. The Leeds and Northrup Company also co-operated fully by constructing and loaning electrical measuring apparatus for experimental work and by finally building in practical form the apparatus that was developed.

Bureau of Standards
Washington, D. C.



Balance room in Research Laboratory, chemist weighing sample of iron for chemical analysis

CHROMIUM

METHOD FOR CHROMIUM AND VANADIUM IN CHROME-VANADIUM STEELS BUREAU OF STANDARDS

Dissolve the sample (2.00 g.) contained in a 300 cc. covered Erlenmeyer flask in 10 cc. of 30% sulphuric acid and 20 cc. of water. Dilute to 200 cc. with boiling water and add from a burette sodium bicarbonate solution (80 g. per 1.) until a permanent precipitate is formed, and then 4 cc. more. Boil one minute, allow to settle and filter rapidly. Wash 4 to 5 times with boiling water. (The filtrate may be used for the determination of the manganese.)

Ignite the residue on the filter paper in an iron crucible and fuse with 10 to 12 times its volume of sodium peroxide. Dissolve the melt by immersing in 100 cc. of water, remove the crucible and destroy any remaining peroxide. (This may be accomplished by heating for one-half hour on the steam bath.)

Filter into a 200 cc. graduated flask and fill up to the mark with water. Acidify 100 cc. of this solution with sulphuric acid and titrate with ferrous sulphate and permanganate. The chromium may be calculated from the number of cc. of permanganate equivalent to the chromium reduction.

Neutralize the remaining 100 cc. of solution with sulphuric acid and then add 3 cc. more of sulphuric acid (sp. gr. 1.84). Heat the solution to boiling and reduce in a Jones reductor allowing the reduced solution to flow into a ferric alum solution contained in the receiving flask. Remove the receiving flask, decolorize the ferric iron by adding a few cc. of phosphoric acid and titrate hot with potassium permanganate. Subtract from the permanganate used one-third the number of permanganate equivalent to the chromium reduction in the first 100 cc. aliquot. The remainder represents the amount of permanganate required to oxidize the vanadium from V_2O_2 to V_2O_5.

In passing through the Jones reductor the chromium is reduced to CrO and the vanadium to V_2O_2. These react with the ferric alum more or less completely reducing some of it to the ferrous condition. The permanganate oxidizes the chromium to the Cr_2O_3 condition, the vanadium to V_2O_5 and the iron to the ferric condition.

DETERMINATION OF CHROMIUM
BY BISMUTHATE METHOD

This method is based on the fact that chromium and manganese are oxidized by sodium bismuthate in either nitric acid, sulphuric acid, or a mixture of nitric and sulphuric acids.

Nitric acid alone is generally used, and only in rare instances will it be necessary to add sulphuric acid to facilitate the solution of the metal. Some manganese is oxidized to permanganic acid, which is decomposed by boiling, forming nitrate of manganese and manganese dioxide.

The manganese dioxide is removed by filtration through asbestos, washing the asbestos well with a 3% solution of nitric acid. If any chromium is present it will be indicated by a yellow color in the filtrate.

Dissolve 3 grams of the sample in a mixture of 70 cc. of water and 30 cc. of nitric acid, (1.42 Sp. Gr.). Boil until metal is in solution. Cool slightly and add 2 grams of sodium bismuthate, taking care to wash all bismuthate from the neck of the flask.

Boil for 15 minutes, or until permanganic acid is decomposed. Filter with suction on asbestos supported by a tuft of glass wool in a 3″ glass funnel. Wash with 3% nitric acid. Cool to tap water temperature and dilute to 500 cc. with distilled water. Add a measured excess of ferrous ammonium sulphate solution until free from yellow tints. Titrate the excess with standard permanganate to faint pink color that persists for 30 seconds.

Titrate with permanganate 50 cc. of ferrous ammonium sulphate containing the same amount of acid and water as the test.

The amount of ferrous ammonium sulphate oxidized by the chromic acid and measured in terms of permanganate is multiplied by the iron factor x .31, or 1 cc. of tenth normal potassium permanganate equals .00173 gram of chromium.

The ferrous ammonium sulphate is prepared by dissolving 50 grams of the salt in 2 liters of 10% by volume of sulphuric acid. When this strength solution is used, 1 cc. will equal about ½ cc. of tenth normal permanganate.

The following table indicates the accuracy of the method, showing in some instances a very small percentage of chromium in the presence of a very high percentage of manganese:

Chromium Added	Chromium Found	Manganese
1.44	1.42	.67
1.44	1.48	.01
.37	.37	.38
.33	.35	.55
.29	.31	.75
.24	.26	.95
.12	.11	1.30
.04	.04	1.90

DEMOREST[1] METHOD FOR THE DETERMINATION OF CHROMIUM AND VANADIUM

Dissolve 2 grams of the sample in a 400 cc. flask by a mixture of 12 cc. of strong sulphuric acid and 50 cc. of water, heat the flask until solution is complete, set it off the hot plate and add, very cautiously, 25 cc. of nitric acid, (1.42 Sp. Gr.) The iron is immediately oxidized to the ferric state with the evolution of much nitrous fumes. Heat the solution to boiling until the brown fumes are all driven off, then set the flask off the hot plate and add sodium bismuthate until everything in solution is oxidized and the manganese appears as permanganic acid and does not disappear on shaking. Dilute the solution to 200 cc., add a little more sodium bismuthate, and boil the solution for 20 minutes to decompose the permanganic acid to manganese dioxide. Cool the solution by adding 50 cc. of water and filter through asbestos. Then make the volume up to 300 cc. and cool to tap-water temperature, when it is ready for the chromium titration after the addition of 5 cc. of syrupy phosphoric acid to decolorize the iron.

To titrate, run in $N/100$ ferrous sulphate solution until all chromium and vanadium are reduced. This can be discerned by testing a drop on a white plate with a drop of ferricyanide. If a blue color is obtained enough ferrous sulphate has been added. Add $N/100$ permanganate until a pink color appears which persists on shaking, then add a few more cubic centimeters of $N/100$ permanganate and stir the solution for a minute. Then add $N/100$ ferrous sulphate until the pink just disappears. The total ferrous sulphate minus the permanganate multiplied by 0.0001733 equals the chromium present.

Add to the solution enough ferrous sulphate to reduce the vanadium and to have a considerable excess, mix the solution and add about 1 gram of 20-mesh ignited natural manganese dioxide. Shake the solution until a test with ferricyanide on a white plate shows that all ferrous iron has been oxidized. Then filter through asbestos (using suction) and titrate. Add $N/100$ permanganate until a persistent pink color is obtained, then add several more cubic centimeters and shake the solution for a minute. Now add $N/100$ ferrous sulphate until the pink color just disappears. The permanganate used minus the ferrous sulphate used multiplied by 0.00051 equals the vanadium present.

NOTES ON THE PROCESS. The bismuthate oxidizes the chromium, vanadium and manganese to chromic acid, vanadic acid and permanganic acid. Boiling destroys the permanganic acid and

1 The Journal of Industrial and Engineering Chemistry, December, 1912.

manganese dioxide precipitates and is filtered off. When ferrous sulphate is added, chromic and vanadic acids are reduced to trivalent chromium and quadrivalent vanadium; then when permanganate is added vanadium only is oxidized back and the ferrous sulphate minus the permanganate measures the chromium.

Vanadium is again reduced by ferrous sulphate (not measured), the excess of ferrous sulphate oxidized by manganese dioxide, leaving the vanadium ready to be titrated.

The following are some results obtained by the above method:

PERCENTAGES CHROMIUM

Present	Found
0.078	0.086
0.155	0.160
0.155	0.157
0.233	0.236
0.078	0.078
0.233	0.225
3.100	3.095

A blank must be run on the chromium determination, as a small amount of MnO_2 persists in solution and runs uniformly the same.

DETERMINATION OF COPPER BY COLORIMETRIC METHOD

Dissolve 10 grams of drillings in a mixture of 25 cc., 1.84 sp. gr., sulphuric acid and 250 cc. of distilled water, using a 500 cc. flask. Heat carefully until the borings have dissolved, dilute to 400 cc. with distilled water and add 0.5 gram zinc sulphide*, cork flask for a few minutes, filter on an 11 cm. paper, wash the residue with hydrogen sulphide water, open paper against side of funnel, add 20 cc. of hot nitric acid, 1.18 sp. gr., to the residue on the paper, allowing the solution to run into the flask in which the borings had been dissolved.

Wash the paper with 2% nitric acid solution, evaporate the filtrate to about 15 cc., remove from hot plate and add ammonia water (1:3) just sufficient to precipitate the ferric hydroxide.

Filter into a 100 cc. Nessler tube and wash with hot water. The presence of copper will be indicated by the blue color of the filtrate from the ferric hydroxide. To another Nessler tube add about 50 cc. of distilled water and 5 cc. of (1:3) ammonia water. Then add from a burette a standard copper solution until the colors match when diluted to the same volume.

Modification. It is sometimes found convenient to modify the method for determining copper. After determining the sulphur by the evolution method the hydrochloric acid solution can be used for the determination of this element as follows:

The solution from the determination of sulphur is neutralized with ammonia until there is a slight precipitate of ferrous hydroxide. Acidify with 5 cc. of hydrochloric acid, heat to boiling point, add 0.5 gram zinc sulphide, when dissolved dilute to 400 cc. with distilled water, cork flask for a few minutes, and filter rapidly on an 11 cm. paper. Wash with hydrogen sulphide water and finish the determination as previously described.

Caution The success of the colorimetric method for determining copper depends upon carefully following each detail. Sometimes a green color will be obtained instead of a blue. This is usually due to the use of too much ammonia and can be corrected by acidifying the green solution with dilute sulphuric acid (1:1) and then making the solution faintly ammoniacal.

* The use of zinc sulphide was suggested to us by W. F. Clark, Dunston on Tyne, England.

Standard Copper Solution. The standard copper solution can be prepared by dissolving 7.856 grams of crystallized copper sulphate in about 200 cc. of distilled water and 10 cc. of nitric acid, 1.42 sp. gr., and diluting to 2 liters. This solution can also be prepared by dissolving 2 grams of copper in 20 cc. of dilute nitric acid, and diluting to 2 liters. Each cubic centimeter represents 0.01 per cent copper when using 10 grams for analysis.

DETERMINATION OF COPPER BY IODIDE METHOD

The colorimetric method for determining copper is not sufficiently accurate when this element is in excess of .15 per cent. When such is the case proceed as outlined for the determination of this element by the colorimetric method to the point where dilute ammonia is added to precipitate the ferric hydroxide. Instead of filtering into a 100 cc. Nessler tube use a 250 cc. beaker for this purpose.

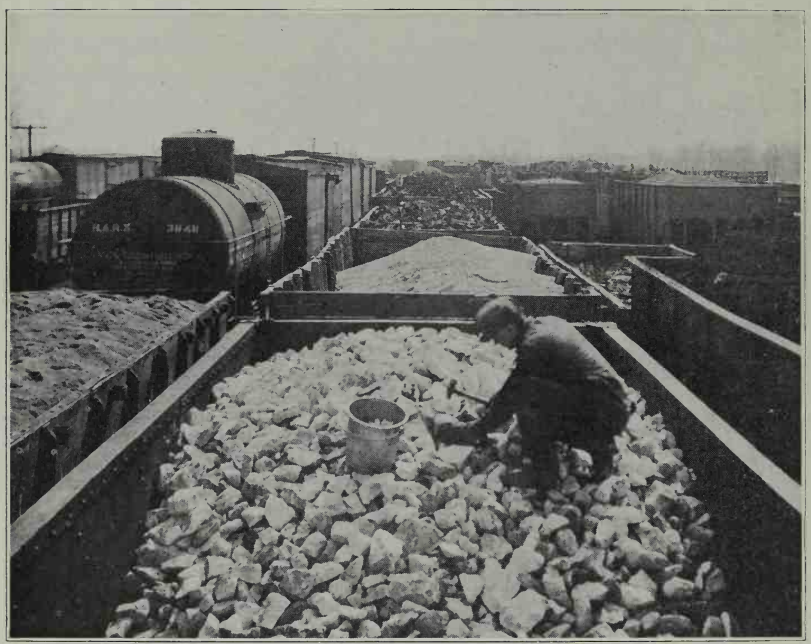
After washing a few times with hot water dissolve the ferric hydroxide with hot dilute hydrochloric acid, allowing the solution to run into the flask in which the original precipitation was made, wash a few times with hot water, add ammonia water 1:3 just sufficient to precipitate the ferric hydroxide and leave a very slight excess of ammonia present, heat to boiling, filter, and allow the filtrate to flow into the 250 cc. beaker containing the major portion of the copper.

Add 5 cc. of sulphuric acid, 1.84 sp. gr., evaporate on hot plate to dense fumes, cool, add 20 cc. of water, make slightly alkaline with ammonia, boil off excess ammonia then neutralize with glacial acetic acid adding 5 cc. in excess, cool and add 5 grams of potassium iodide crystals, then a few cubic centimeters of starch solution, and titrate with $N/20$ sodium thiosulphate solution to the disappearance of the blue color. (12.4 grams per liter, 1 cc. thiosulphate equals .00318 gram copper).

The thiosulphate can be conveniently standardized by using 25 cc. of the standard copper sulphate solution described in the Colorimetric Method for determining copper, adding the same reagents, and titrating under the same conditions as described in the regular method.

If preferred the thiosulphate can be standardized by dissolving 5 grams of potassium iodide in 500 cc. of water, adding 25 cc. concentrated hydrochloric acid, then exactly 25 cc. of standardized $N/10$ potassium bichromate solution. Add starch solution and titrate with the thiosulphate solution to the disappearance of the blue color. Each cc. of $N/10$ potassium bichromate solution equals .00636 gram copper.

Standard Starch Solution. The starch solution used in this method is prepared as described under the determination of sulphur in iron and steel, Page 191.



Limestone is used in Open Hearth furnaces for fluxing the impurities in the manufacture of ARMCO Ingot Iron products. This shows method of sampling each carload received

DETERMINATION OF HYDROGEN

The method for determining hydrogen by heating the metal in a partial vacuum and measuring the liberated hydrogen is very laborious. The hydrogen existing as ammonia would not be estimated by the above method.

Our method is based on the fact that hydrogen is liberated by heating the metal to a red heat in an atmosphere of oxygen. The hydrogen is oxidized to water, which is absorbed in phosphoric anhydride.

The apparatus used consists of a 12″ gas blast furnace for burning the sample, and a 12″ electric furnace for purifying the oxygen gas.

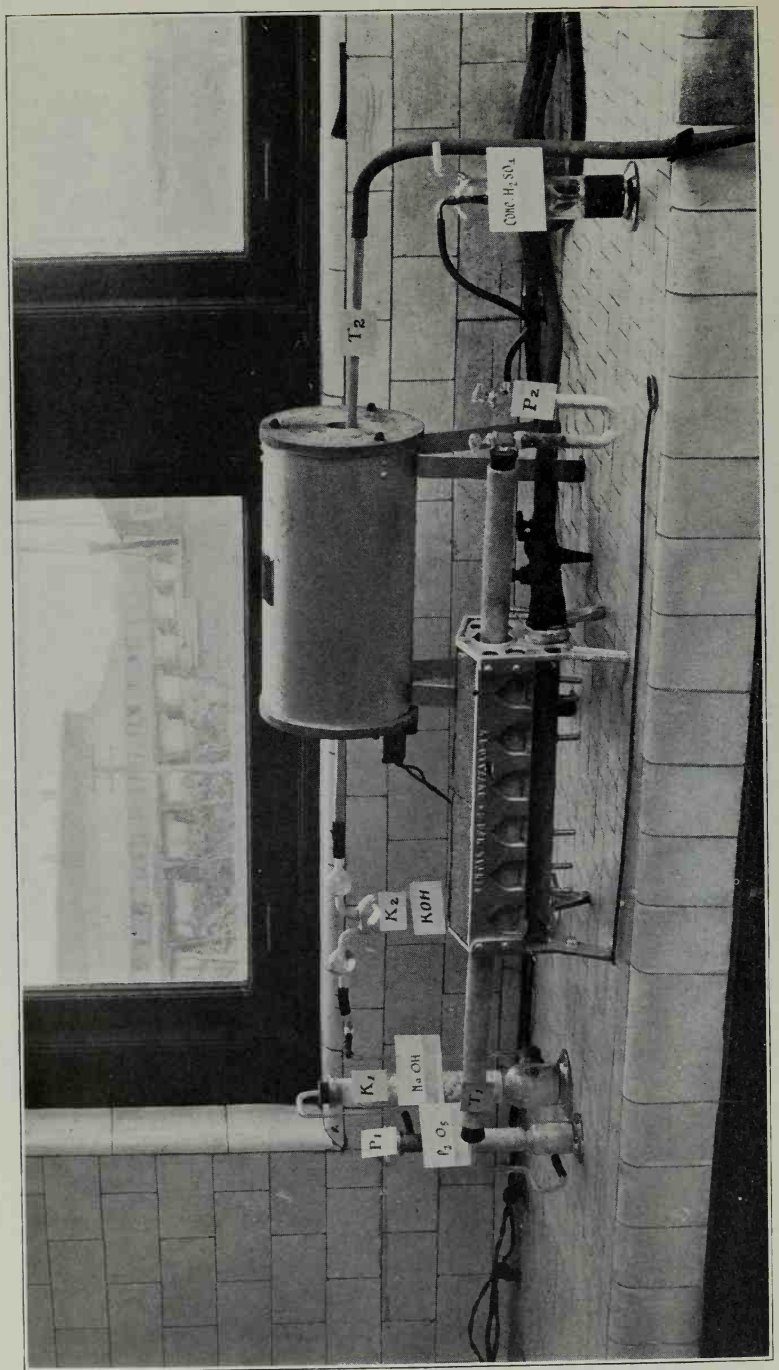
The oxygen gas is passed through a ¼″ silica tube (T-2) at the rate of 25 cc. per minute. This furnace is heated to about 750° C., which is sufficient to purify the oxygen gas. The impurities are absorbed by passing the gas first into a solution of caustic potash (K-2) and then through a bottle containing caustic soda (K), and finally through a tube containing phosphoric anhydride opened up in glass wool (P-1).

The purified oxygen gas then enters the ⅞″ silica tube (T-1) where it combines with the hydrogen which is liberated from the metal, thus forming water which is absorbed in a 4″ glass stopcock U tube containing phosphoric anhydride opened up with glass wool (P-2). This weighed 4″ U tube is connected with the ⅞″ silica tube (T-1), after the sample has been placed in the combustion tube. The weighed U tube is then connected with bottle containing sulphuric acid (Conc.).

The combustion tube is heated to a temperature not to exceed 800° C., as a higher temperature and a higher rate of oxygen than prescribed may generate enough heat to damage the apparatus. The oxygen passes through the entire apparatus at the rate of 25 cc. per minute for 30 minutes, and from 10 to 40 grams of drillings are used for a determination.

After the test is complete there should be some metal which has not been oxidized, as it has been found unnecessary to oxidize all of the metal in obtaining accurate results.

After having ignited the sample for 30 minutes, the weighed U tube (P-2) is disconnected, and connected with an aspirator for the purpose of replacing the oxygen in the tube with dry air.



Apparatus for the determination of Hydrogen in Iron and Steel

A suitable aspirator for this purpose consists of a one gallon aspirator bottle filled with water connected with absorbing tubes as shown in photograph of apparatus for the determination of oxygen and carbon monoxide in iron and steel on page 158.

The increased weight of the U tube which is due to the water which has been absorbed is multiplied by .11190 then by 100, and divided by the weight of sample taken. This gives the percentage of hydrogen in the metal.

Chemist making a calorimeter test on coal to determine its heating value

GRAVIMETRIC DETERMINATION OF IRON

Dissolve about 1 gram[1] of the sample, accurately weighed, in a 600 cc. Jena glass beaker[2], on the water bath, using 25 cc. of a 10% solution of hydrochloric acid.

When solution is complete, add 200 cc. of distilled water, heat on water bath to about 80° C., and pass a moderate current of hydrogen sulphide gas through the solution for 20 minutes.

Remove from water bath, add 200 cc. more of cold distilled water, and continue the stream of hydrogen sulphide for another 20 minutes, or until the solution is cold. Filter from the precipitate and wash thoroughly with hydrogen sulphide water containing a small amount of hydrochloric acid[3], collecting the filtrate in an 800 cc. Jena glass beaker[2].

Test the residue for iron[4]. Evaporate the filtrate on the water bath until the volume is reduced to about 200 cc. and all the hydrogen sulphide is driven off. Then add 5 cc. of concentrated nitric acid and 10 cc. of concentrated hydrochloric acid[5], and heat to about 90° C., on the water bath and add a slight excess of warm dilute ammonia[6].

Allow the precipitate to settle, decant through a 15 ctm. ashless filter, transfer the precipitate to the filter[7] and wash with boiling water until 5 cc. of the washings give no opalescence with silver nitrate[8]. (Collect the filtrate and the first washings in a clean 800 cc. beaker and reserve for determination of manganese).

Dry the ferric hydroxide precipitate at 95–100° C[9]., and then separate as perfectly as possible from the filter paper, in a room free from draught, placing the dry ferric hydroxide in a small porcelain dish on a white glazed paper. Cover the porcelain dish with a watch glass and then ignite the filter paper in a weighed porcelain crucible.

Transfer the precipitate from the porcelain dish to the crucible; cover with a platinum cover and ignite for 10 minutes over a Bunsen burner; remove the cover, incline the crucible slightly and ignite for another 10 minutes. Place in desiccator, cool and weigh. Repeat the ignition until the weight remains constant, taking care not to heat more than a few minutes at a time and not at too high temperature[10].

In the meantime evaporate the filtrate for the determination of manganese, to about 200 cc. Add ammonia, heat to boiling and precipitate the manganese with a saturated solution of bromine. Boil

for a few minutes, filter on a small ashless filter, ignite and weigh as Mn_3O_4. This weight subtracted from the weight of Mn_3O_4 in the sample, calculated from the total manganese, gives the amount of Mn_3O_4 in the ferric oxide.

To determine silica in the ignited precipitate, transfer this to a small platinum dish and digest with concentrated hydrochloric acid on the water bath and evaporate to dryness[11]. Redissolve, dilute with hot water and filter from the silica on a small ashless filter. Wash with hot dilute hydrochloric acid and hot water until the silica is free from iron.

Ignite in a platinum crucible, cool and weigh, and then evaporate with 2 drops of sulphuric and 1 cc. of hydrofluoric acid; ignite, cool and weigh. The difference between the two weighings then represents the amount of silica in the ferric oxide[12].

The total amount of silica, manganese oxide, chromic oxide, phosphoric acid and alumina (calculated from analysis), subtracted from the weight of impure ferric oxide, gives the weight of pure Fe_2O_3, which contains 69.94% iron (Fe = 55.84).

NOTES ON GRAVIMETRIC DETERMINATION OF IRON

(1). By employing a power-driven centrifuge of large capacity the washing of precipitate can be perfectly and quickly performed largely by decantation, thereby enabling the operator to use a sample as large as 5 grams.

(2). Beakers, funnels, watch glasses and glass rods must be thoroughly cleaned with warm concentrated hydrochloric acid before use, in order to prevent any foreign iron from entering the solutions.

(3). The presence of acid is necessary to secure a perfect removal of iron from the residue and the filter paper. The hydrogen sulphide removes the following elements: Silver, lead, mercury, gold, platinum, tin, antimony, arsenic, copper, cadmium, bismuth, molybdenum, tellurium, selenium, germanium, iridium, osmium, palladium, rhodium, ruthenium, tungsten, vanadium.

(4). If great accuracy is desired, the analysis should be rejected whenever more than traces of iron are detected in the sulphide residue.

(5). The nitric acid is added to oxidize the iron, and the hydrochloric acid to secure sufficient ammonium chloride to keep zinc,

cobalt, nickel and part of the manganese in solution. In nickel steels the precipitation should be repeated several times.

(6). A large excess of ammonia is objectionable, as it causes some of the iron to become colloidal.

(7). Ferric hydroxide adheres to the beaker and the glass rod. In order to remove this quantitatively, a few drops of concentrated nitric acid are introduced into the beaker and by means of the glass rod all ferric hydroxide is easily brought in solution. After dilution with water the iron is reprecipitated with ammonia and transferred to the filter. Nitric acid is used in order to prevent introduction of chlorides. (See 8).

(8). When iron is precipitated with ammonia, small amounts of basic iron salts are always thrown down with the hydroxide. The amount and the composition of the basic salts vary according to the conditions. Thus in a solution of sulphate of iron larger amounts of basic salts are formed than from solutions of ferric nitrate or chloride. In a cold solution more basic salts are formed than when the solution is nearly boiling, and in addition the ferric hydroxide tends to assume a colloidal state, especially in the presence of a large excess of ammonia. Basic chloride of iron is volatile on ignition, hence the necessity of eliminating chlorides. Warm water decomposes chloride of iron, leaving the hydroxide free from chlorine.

(9). The filter paper will become brittle if heated at a temperature above 100° C. Dry at least 10 hours. Heat gradually in crucible.

(10). When ferric oxide is ignited at too high a temperature, some magnetic oxide of iron (Fe_3O_4) is always formed, causing low results. The formation of magnetic oxide takes place much more readily when the ignition is performed in a platinum crucible. A convenient arrangement consists of placing a small porcelain crucible within a covered platinum crucible, whereby the contact with platinum is avoided and the danger of overheating greatly reduced; at the same time the disadvantage of using a porcelain crucible alone is overcome.

(11) If the oxide has been heated to a high temperature it is difficult to dissolve in concentrated hydrochloric acid. To secure solution in a reasonable length of time in cases when the oxide has been overheated, it is advisable to grind the oxide carefully in an

agate mortar and then ignite it for a minute, reweigh and determine the silica on this portion and from the result calculate the silica in the original amount of oxide.

(12). In all exact gravimetric determinations of iron, allowance must be made for silica in the ferric oxide. Most steel and iron contain silicon, and considerable amounts are always dissolved from the glassware during the chemical operations. Glass is readily attacked by warm ammonia. Part of the silica is undoubtedly derived from the ammonia which ordinarily has been in contact with common glass. Most of the phosphorus is evolved during the solution in hydrochloric acid. If, however, the sample contains more than a few thousandths of 1% of phosphorus, the ferric oxide should be analyzed for phosphorus. This can be done in the filtrate from the determination of silica, and the amount found, figured to phosphoric anhydride, added to the other impurities.

MANGANESE

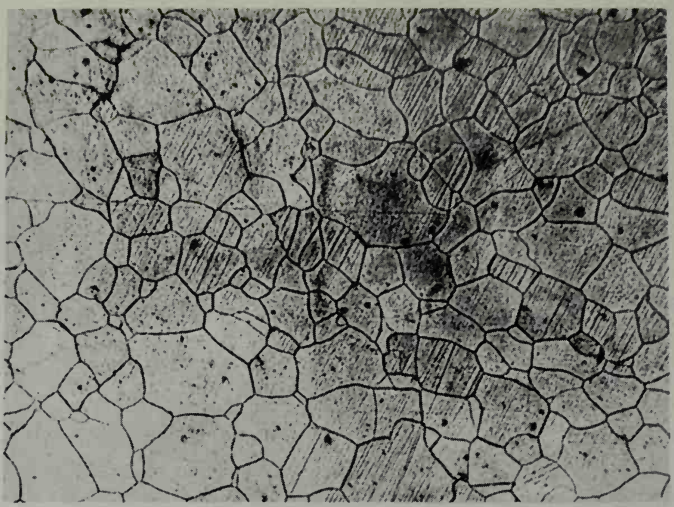
PERSULPHATE METHOD FOR THE DETERMINATION OF MANGANESE

This method is of more value in the determination of manganese in steel than in pure American Ingot Iron. However, with proper care accurate results can be obtained on American Ingot Iron. The method is as follows:

Dissolve .5 gram of the sample in a 250 cc. Erlenmeyer flask using 25 cc. (1.20 Sp. Gr.) nitric acid, heat on water bath until brown fumes are gone. Remove flask from the water bath and add 40 to 50 cc. $N/100$ silver nitrate, return flask to water bath and heat to 50 to 60 degrees C. Add about 2 grams of crystallized ammonium persulphate and maintain the solution at 50 to 60 degrees C., for a few minutes.

Cool, dilute with 100 cc. distilled water and titrate with sodium arsenite to pale green color.

It is necessary to keep the temperature between 50 and 60 degrees C. during the few minutes required for the perfect oxidation of the manganese, otherwise results will be erratic, especially when analyzing American Ingot Iron.



AMERICAN INGOT IRON

Sulphur	.032
Phosphorus	.008
Carbon	.011
Manganese	.017
Silicon	trace
Copper	.030
Oxygen	.025
Nitrogen	.003
Iron	99.874

Microstructure and Analysis

DETERMINATION OF MANGANESE
BISMUTHATE METHOD

This method was perfected by Professor D. J. Demorest, of the Ohio State University and is substantially as follows:

Dissolve 1 gram of the sample in 30 cc. of Nitric Acid (Sp. Gr. 1.13) and boil until brown fumes disappear. After cooling somewhat one-half gram of sodium bismuthate is added, a little at a time, until the resulting permanganic acid or manganese dioxide persists after a few minutes boiling. Now add 3 cc. of a 5% solution of potassium nitrite to reduce the manganese compounds, and boil the solution a few minutes to expel the nitrous fumes.

Cool to tap water temperature and when cold add sodium bismuthate, a little at a time, while the solution is shaken, until about ½ gram has been added.

After settling a few minutes the solution is filtered by suction through asbestos on glass wool contained in a 3″ funnel.

The filter is well washed with a 3% solution of nitric acid prepared from colorless acid, and containing a small amount of sodium bismuthate in suspension. The permanganic acid is then titrated with standard sodium arsenite solution until the pink tinge just disappears. There should not be a brownish color at the end. If there is, it indicates insufficient acid.

The sodium arsenite is prepared by dissolving 2 grams of arsenious acid in a hot solution of sodium carbonate, using sufficient sodium carbonate to completely dissolve the arsenious acid. It is then filtered through paper and diluted to 2 liters. One cc. is approximately equal to .00025 gram of manganese. The solution is standardized against steel of known manganese content.

Important Details

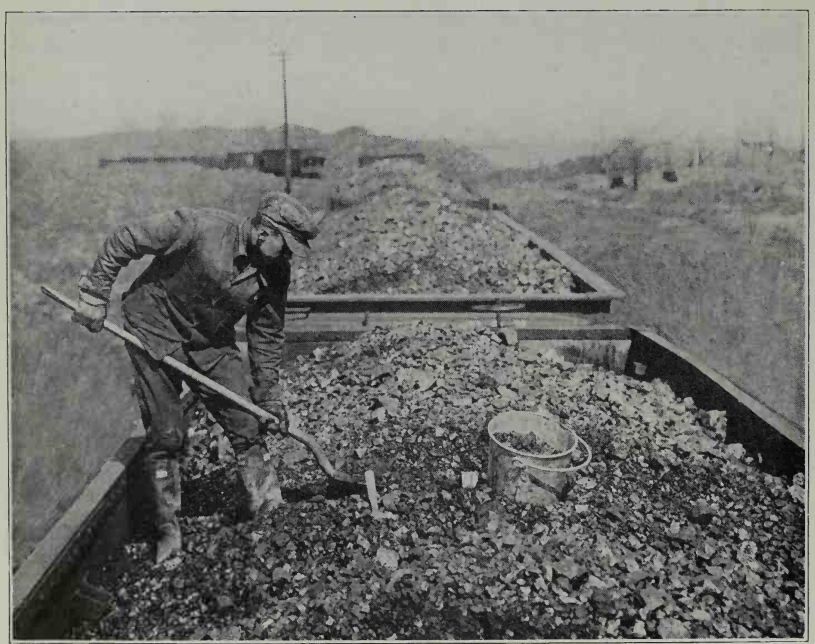
(1). Do not filter immediately after the addition of sodium bismuthate, but let stand at least one minute.

(2). Keep the asbestos filter clean and level across the top.

To clean: Wash filter with hot concentrated nitric acid, then run through the filter a strong solution of potassium permanganate and finally wash clean with the prepared wash water solution.

(3). Use care in preparing the wash water solution, being sure that it is 3% nitric acid in strength and contains a small amount of free sodium bismuthate in suspension.

A modification of this method is described by P. L. Robinson in Chemical News 119, 187-8 (1919). The modification consists of adding 10 cc. of ammonium persulphate solution (120 g. per liter) for the preliminary oxidation of the carbonaceous matter. Do not remove the assay from the plate after solution, but immediately after the brown fumes have cleared add the ammonium persulphate; then boil for 10 minutes to decompose the excess of ammonium persulphate, cool, oxidize with sodium bismuthate, filter, and titrate in the usual manner.

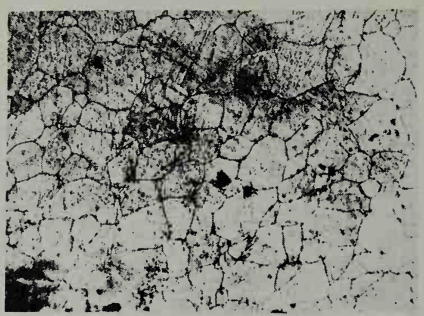


Taking samples of coal by the Trench Method. Three trenches being dug in each car of coal received. The sample which is taken is thoroughly ground for chemical analysis. The control of the quality of coal which enters into all of our metallurgical practice is one of the reasons for the regularity and uniformity of the products which we manufacture

DETERMINATION OF MANGANESE BY COLOR

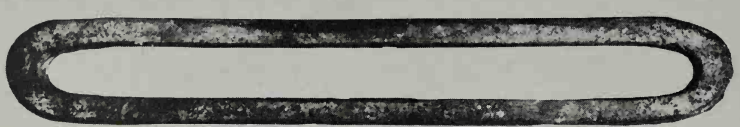
Dilute the solutions from color carbon determination to 30 or 40 cc. Remove 10 cc. from each with a pipette, place in 10″x1″ test tubes and add to each 3 cc. of nitric acid (Sp. Gr. 1.18). Bring to boil and add ½ gram of lead peroxide (free from manganese) to each and boil vigorously for 3 minutes.

Cool and pour into 15 cc. centrifuge tubes and separate the undissolved lead peroxide by centrifuging a few minutes. Decant clear solution into comparison tubes and dilute until colors match. If no centrifuge is available the lead peroxide can be filtered out on an alundum crucible or on an ignited asbestos filter of the Gooch or cone type.



NEWBURYPORT LINK

Sulphur	.014
Phosphorus	.023
Carbon	.040
Manganese	.008
Silicon	.028
Copper	trace
Oxygen	.027
Nitrogen	.003
Iron	99.867



After 100 years service Microstructure and Analysis

TEST TO INDICATE WHETHER METAL IS IRON OR STEEL

In making this test without the use of a balance the following table can be used when metal is in sheet form and the gauge is known. These weights represent the number of grams in 1 square inch:

Gauge	Grams Sq. Inch	Gauge	Grams Sq. Inch	Gauge	Grams Sq. Inch
12	14.00	17	7.10	22	4.00
13	12.00	18	6.40	23	3.61
14	10.00	19	5.66	24	3.20
15	9.00	20	4.82	25	2.80
16	8.00	21	4.41	26	2.41

As an illustration, suppose we have 16-gauge material. A square inch weighs 8 grams, hence a strip ⅛″x1″ weighs approximately 1 gram, or ¼″x½″ would also equal 1 gram. If the sheet is galvanized the coating need not be removed before making the test.

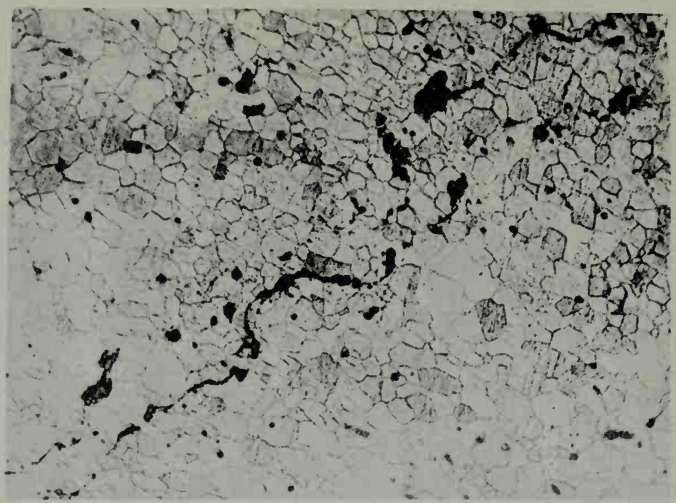
Take equal portions of American Ingot Iron and sample to be tested, equal to ½ gram, and place in separate 10″x1″ test tubes. Add to each tube 15 cc. [1] of dilute nitric acid, 1.18 specific gravity[2]. Place a test tube in holder, using care to incline the tube away from spectators while being heated with an alcohol lamp. The metal will disappear in a few minutes, but continue heating until no more brown fumes are given off. Allow solution to cool and heat the other tube in same manner and cool. The solutions can be compared at this point, the darker one containing the highest percentage of carbon.

To each test add ½ gram sodium bismuthate[3] and agitate for a few minutes. Then add sufficient water to half fill the tubes and mix thoroughly. Allow the tubes to rest for several minutes until the undissolved sodium bismuthate settles. By comparing the clear solutions, American Ingot Iron will show a light pink color, while steel will yield a purple color due to manganese present.

1 If no graduate is available, the volume of acid can be estimated by noting the depth of 1″ diameter tube; each inch is equal to about 10 cc.

2 Nitric Acid of 1.18 specific gravity can be prepared by adding 1 part 1.42 specific gravity nitric acid to 2 parts water.

3 The amount of sodium bismuthate that can be placed on a dime represents about ½ gram.



BESSEMER STEEL

Sulphur	.050
Phosphorus	.100
Carbon	.120
Manganese	.480
Silicon	trace
Copper	.010
Oxygen	.025
Nitrogen	.010
Iron	99.205

Microstructure and Analysis

MOLYBDENUM

THE DETERMINATION OF MOLYBDENUM (AND COPPER) IN STEEL

BUREAU OF STANDARDS

Dissolve enough steel to give .06 to .1 g. molybdenum, in 1:3 nitric acid. When solution is complete, add 10 cc. of sulphuric acid and evaporate to fumes. Cool and dilute. If the residue is light colored and evidently silica no filtration is required.

In case the residue is dark colored or indicates tungstic acid, filter it off, ignite it carefully in platinum, fuse the residue with sodium carbonate, thoroughly extract the melt with water, add two grams tartaric acid to the filtered water extract, acidify to two per cent by volume sulphuric acid and saturate with hydrogen sulphide. Digest one hour at approximately 50° C., filter off any dark sulphide and wash with a one per cent by volume sulphuric acid saturated with hydrogen sulphide and containing a little tartaric acid. Dissolve the precipitate in aqua regia and add the resultant solution to the solution of the sulphides which is obtained as described below.

Dilute the main solution to 200 cc., add 5 grams tartaric acid and adjust the acidity to ½% sulphuric acid (by volume). Pass in hydrogen sulphide until the iron is reduced, the molybdenum (and copper) is precipitated, and the solution is saturated with gas. Digest at 50°–60° for one hour or longer. Filter, preferably on a Gooch, and wash with the wash water specified above. Dissolve the precipitate in aqua regia, unite with any recovery obtained as above and reserve the solution.

Occasionally the filtrate from the precipitated sulphides contains some molybdenum which was not precipitated on account of reduction of the molybdenum. It is desirable to test this filtrate as follows: boil out most of the hydrogen sulphide, oxidize the iron and molybdenum by means of bromine water, boil out excess bromine and again proceed with the hydrogen sulphide precipitation. In case molybdenum sulphide is indicated it is to be recovered as in the regular procedure and added to the reserved solution.

The reserved solution will contain the molybdenum and copper originally present in the steel and may contain a little iron. Treat this solution with 5 cc. of sulphuric acid and evaporate to fumes. Dilute and in case the solution is colored by reduced molybdenum, oxidize with a little permanganate solution. Then add a 10% solution of sodium hydroxide until in slight excess. Boil, filter, and wash with hot 1% sodium hydroxide solution. The insoluble contains the copper together with a little iron, and the copper may be determined electrolytically.

The filtrate contains the molybdenum and this is most conveniently determined as follows: acidify the solution, add sulphuric acid until it contains 3% by volume, warm the solution and reduce in a Jones' reductor containing a solution of ferric alum and phosphoric acid in the receiver. The molybdenum is thus reduced to the trivalent condition in the reductor and is partially oxidized by the ferric sulphate in the receiver with the formation of an equivalent amount of ferrous sulphate. Titrate the resultant solution with tenth normal permanganate. The molybdenum and ferrous sulphate are oxidized to their higher valencies by the permanganate and the calculations are based on complete reduction to Mo_2O_3 and subsequent oxidation to MoO_3.

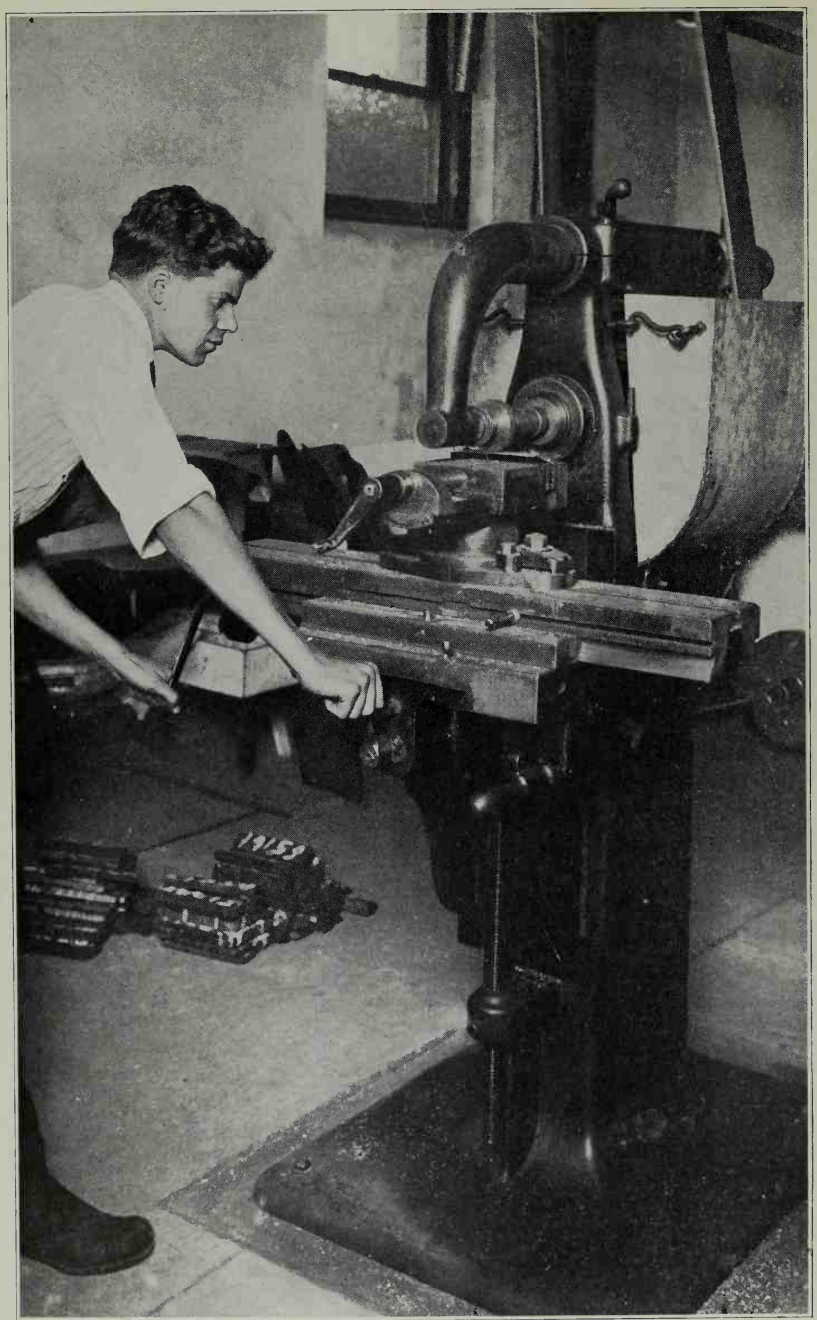
BRUNCK'S[1] GRAVIMETRIC DETERMINATION OF NICKEL

Dissolve 1 gram of steel or iron in a 150 cc. Erlenmeyer flask with the use of 30 cc. of dilute nitric acid, (1.20 Sp. Gr.). Boil until brown fumes are expelled. Place 5 grams of powdered citric acid in an 800 cc. beaker. Add the solution in the Erlenmeyer flask to the dry citric acid, wash the flask thoroughly with water, and make the volume in the beaker up with water to 300-500 cc., depending upon the amount of nickel present. The higher the nickel the more water necessary. Make faintly alkaline with ammonia, then acid with acetic acid.

Note: Acetic acid is preferred to hydrochloric on account of a more perfect separation of nickel from manganese when manganese is present.

The faintly acetic acid solution is heated to near the boiling point, the beaker is removed from the source of heat, and from 15 to 25 cc. (depending upon the amount of nickel present) of a 1% alcoholic solution of dimethylglyoxime added. The solution is then made faintly alkaline with dilute ammonia, the nickel being precipitated as scarlet nickel glyoxime. The solution is kept hot for about 1 hour and filtered on a weighed Gooch crucible, washed thoroughly with hot water, and dried at 110-120° C. for 45 minutes. This scarlet precipitate contains 20.31% nickel. If the percentage of nickel is low we use 3 grams of the sample which we dissolve in 50 cc. of nitric acid (Sp. Gr. 1.20). We also use 15 grams of citric acid. The presence of chromium or cobalt does not interfere with the precipitation.

1 Stahl u. Eisen; 28 (1) p. 331; 1908.



View of milling machine showing milling of sheet bars for the determination of oxygen and carbon monoxide

DETERMINATION OF NICKEL IN STEEL
TITRATION METHOD

The determination of nickel in steel can be finished in less than 25 minutes with the use of the method devised by the Buckeye Steel Casting Company.

Dissolve 1 gram of steel or iron in a 150 cc. Erlenmeyer flask with the use of 30 cc. of dilute nitric acid, (1.20 Sp. Gr.). Boil until brown fumes are expelled. Wash the solution into an 800 cc. beaker, add 20 cc. of Citric Acid solution. Make faintly alkaline with ammonia, and acidify slightly with acetic acid. (Acetic acid is preferred to hydrochloric on account of a more perfect separation of nickel from manganese when manganese is present.) The faintly acetic acid solution is heated to near the boiling point, the beaker is removed from the source of heat, and from 15 to 25 cc. (depending upon the amount of nickel present) of dimethylglyoxime solution added. The nickel being precipitated as scarlet nickel glyoxime.

Bring to boil and filter immediately washing the precipitate from the filter with the use of hot water into a 250 cc. beaker. Dissolve the nickel glyoxime in 20 cc. of aqua regia, bring to boil in order to decompose the glyoxime. Dilute with about 50 cc. of cold water, make faintly ammoniacal and cool in ice water. Make solution up to 150 cc. with cold water, add 10 cc. of potassium iodide solution, then 1 cc. of silver-nitrate solution and titrate with potassium cyanide solution (be sure to do this under the hood on account of the poisonous nature of the hydrocyanic acid) until the turbidity disappears and the solution clears.

The potassium solution cyanide is standardized with the use of Bureau of Standards nickel steel, or with the use of .2 to .3 grams of nickel ammonium sulphate which has been added to one gram of iron or steel free from nickel and analyzed according to the above method. The following solutions are used in the determination of nickel by this method:

Potassium Iodide

Dissolve 8 grams of potassium iodide in one liter of distilled water.

Silver Nitrate Solution

Dissolve 5 grams of crystallized silver nitrate in one liter of distilled water.

Potassium Cyanide Solution

Dissolve 13 to 14 grams of potassium cyanide in water and when in solution add 5 grams of potassium hydroxide which has also been dissolved in water and dilute the solution to one liter. Potassium cyanide containing sulfide cannot be used; as it forms a precipitate of silver sulfide which is not dissolved by potassium cyanide.

Dimethylglyoxime Solution

Dissolve 20 grams of dimethylglyoxime in 1300 cc. of concentrated ammonia, make the solution up to 2 liters with the use of 700 cc. of distilled water.

Aqua Regia Solution

As this acid decomposes upon standing it is desirable to make up fresh solutions each day; mixing 80% concentrated nitric acid and 20% concentrated hydrochloric acid.

Citric Acid Solution

Dissolve 600 grams of citric acid in one liter of distilled water.

In standardizing the Potassium Cyanide Solution the blank produced from 1 cc. of silver nitrate solution should be deducted before determining the strength of the potassium cyanide, and this blank amounting usually to .3 cc. should be deducted from each determination.

It will be satisfactory to determine the blank using the same amount of water (made ammoniacal) as the volume of a determination (200 cc.) using about 10 cc. of potassium iodide and 1 cc. of silver nitrate, titrating with potassium cyanide until the solution clears.

NITROGEN

DETERMINATION OF NITROGEN

We use the Allen method perfected by Professor J. W. Langley. This method is based on the reaction by which the combined nitrogen in iron or steel is estimated as ammonia by the solution of the metal in hydrochloric acid.

The reagents required are:

Hydrochloric Acid of 1.1 specific gravity, free from ammonia, which may be prepared by distilling pure hydrochloric acid gas into distilled water free from ammonia. To do this, take a large flask fitted with a rubber stopper carrying a separatory funnel tube and an evolution tube. Place in the flask strong hydrochloric acid, connect the evolution tube with a wash bottle connected with a bottle containing the distilled water. Admit strong sulphuric acid free from nitrous acid to the flask through the funnel tube, apply heat as required, and distil the gas into the prepared water.

Test the acid by admitting some of it into the distilling apparatus, described further on, and distilling it from an excess of pure caustic soda, or determine the amount of ammonia in a portion of hydrochloric acid of 1.1 specific gravity, and use the amount found as a correction.

Solution of Caustic Soda, made by dissolving 300 grams of fused caustic soda in 500 cc. of water, and digesting it for 24 hours at 50° C., on a copper-zinc couple prepared by rolling together about 6 square inches each of zinc and copper foil.

Nessler Reagent. Dissolve 35 grams of potassium iodide in a small quantity of distilled water, and add a strong solution of mercuric chloride little by little, shaking after each addition, until the red precipitate formed dissolves. Finally the precipitate formed will fail to dissolve; then stop the addition of the mercury salt and filter. Add to the filtrate 120 grams of caustic soda dissolved in a small amount of water, and dilute until the entire solution measures 1 liter. Add to this 5 cc. of a saturated aqueous solution of mercuric chloride, mix thoroughly, allow the precipitate formed to settle, and decant or siphon off the clear liquid into a glass-stoppered bottle.

Standard Ammonia Solution. Dissolve .0382 gram of ammonium chloride in 1 liter of water. One cc. of this solution will equal .01 milligram of nitrogen.

Distilled water free from ammonia. If the ordinary distilled water contains ammonia, redistil it, reject the first portions coming over, and use the subsequent portions, which will be found free from ammonia. Several glass cylinders of colorless glass of about 160 cc. capacity are required.

The best form of distilling apparatus consists of an Erlenmeyer flask of about 1500 cc. capacity, with a rubber stopper carrying a separatory funnel tube and an evolution tube, the latter connected with a condensing tube, around which passes a constant stream of cold water. The inside tube where it issues from the condenser should be sufficiently high to dip into one of the glass cylinders placed on the working table.

The determination of nitrogen is made as follows: Place 40 cc. of the caustic soda, which has been treated with the copper-zinc couple, in the Erlenmeyer flask, add 500 cc. of water and about 2 grams, 20-mesh zinc to prevent bumping, and distil until the distillate gives no reaction with the Nessler reagent. While this part of the operation is in progress, dissolve 3 grams of the carefully washed drillings in 30 cc. of the prepared hydrochloric acid, using heat if necessary. Transfer the solution to the bulb of the separatory funnel tube, and when the soda solution is free from ammonia, very slowly drop the ferrous chloride solution into the boiling solution in the flask. When about 50 cc. of water has been collected in the cylinder, remove it and substitute another cylinder. Place 1½ cc. of the Nessler reagent in a cylinder, dilute the distillate to 100 cc. with the special distilled water and pour it into the cylinder, containing the Nessler reagent. Take another cylinder, place therein 1½ cc. of the Nessler reagent and 100 cc. of the special distilled water to which 1 cc. of the ammonium chloride solution has been added, and compare the colors of the solutions in the two cylinders.

If the solution in the cylinder containing the ammonium chloride solution is lighter in color than that in the cylinder containing the distallate, place 1½ cc. of the Nessler reagent in another cylinder, pour into it 100 cc. of water containing 2 or more cc. of the ammonium chloride solution, and repeat this operation until the colors of the solutions in the two cylinders correspond after standing about 10 minutes. When about 100 cc. have distilled into the second cylinder, replace it and test as before. Continue the distillation until the water comes over free from ammonia, then add together the number of cc. of ammonia solution used, divide the sum by 3, and each .01 milligram will be equal to .001% of nitrogen in the steel.

OXYGEN
CARBON MONOXIDE

THE DETERMINATION OF OXYGEN AND CARBON MONOXIDE IN IRON AND STEEL

Ledebur[1] about 40 years ago, proposed heating the borings in an atmosphere of hydrogen in order to determine the oxygen content. The method as proposed was too laborious, consequently was not universally adopted. He recommended a preliminary heating of the borings in nitrogen. Up until the time Cushman[2] published his paper on the determination of oxygen and showed that an analysis could be made in less than an hour, very little use had been made of the method as proposed by Ledebur. However, since Cushman's paper appeared, a considerable amount of work has been done on this subject by many chemists.

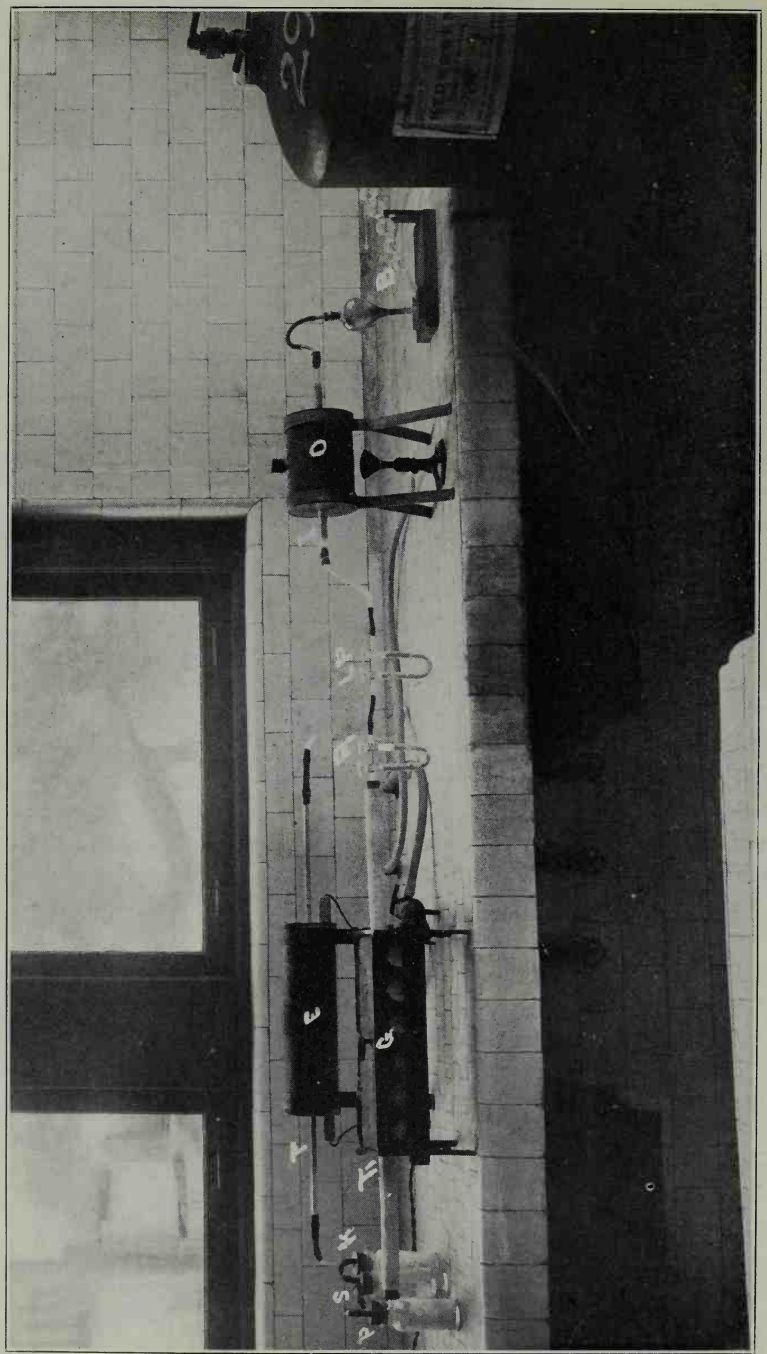
The Ledebur method for determining oxygen is recognized as having its limitations, but where manganese and silicon are low, such as in pure iron, we have found the method of great help in maintaining a uniform product. We have modified the Ledebur method so that we determine the oxygen and carbon monoxide in one operation.

For mill practice where samples can be taken from bars they should first be cleaned from all mill scale or surface oxide with the use of an emery wheel. The sample should then be placed in a milling machine which should be run at very slow speed in order to avoid oxidizing the millings. A light transverse cut should be taken entirely across the bar and the millings discarded in order to remove any oxidized cavities which were not removed by the emery wheel.

The sample should be the average of the entire cross section if possible, as there is some difference in gas content between the interior and exterior portions of bars. The sample must be free from all dirt, and samples should not be ground in the vicinity where a sample is being milled, on account of the danger of contamination from finely divided particles of oxide of iron.

The millings should be removed from the sample by the use of a magnet, and placed in a dry glass stoppered bottle. It is of the utmost importance that millings of uniform size be used for analysis,

1 Leitfaden fur Eisenhütten Laboratories, Eighth Edition, 1908, page 139.
2 Determination of Oxygen in Iron and Steel, by Allerton S. Cushman, Journal of Industrial and Engineering Chemistry, June, 1911.



Apparatus for the determination of oxygen and carbon monoxide in iron and steel

those passing a twenty mesh and remaining upon a forty mesh sieve being selected. The millings should remain in the unstoppered bottle for half an hour in a desiccator containing concentrated sulphuric acid. A 30-gram sample is placed in a ½″x½″x6″ platinum or pure iron boat, (For cast iron use a porcelain boat), which is placed in the ⅞″x30″ silica tube, "T".

In most descriptions for determining oxygen by the Ledebur method, hydrogen is generated by the action of some acid upon zinc contained in a Kipp's generator. It has been found that hydrogen so prepared contains considerable carbon monoxide and carbon dioxide, whereas hydrogen produced by the electrolytic process and stored in tanks is practically free from these two gases, is much easier to handle, and is cheaper.

APPARATUS FOR THE DETERMINATION OF OXYGEN AND CARBON MONOXIDE IN IRON AND STEEL

29—Tank of Electrolytic Hydrogen.
E—Electric Preheating Furnace—850° C.
T—Silicia Tube—¼x30″.
K—Bottle containing sodium hydroxide sticks.
S—Bottle containing concentrated sulphuric acid.
P—Bottle containing phosphoric anhydride on glass wool.
T_1—Silica tube ⅞″x30″ in which is placed boat containing sample.
G—Gas Furnace—Run at 1000° C.
P_1—Absorption Tube containing phosphoric anhydride opened up with glass wool.
LP—U Tube containing phosphoric anhydride used as a trap.
I—Glass tube containing iodine pentoxide.
O—Furnace heated by Bunsen Burner to 150° C.
B—Meyer Bulb containing barium hydroxide solution.

The hydrogen is passed through a ¼″x30″ silica tube "T" contained in an electric furnace "E" heated to 850° C. It is then passed through a bottle "K" containing sticks of sodium or potassium hydroxide, which removes water and carbon dioxide, then through a wash bottle "S" containing concentrated sulphuric acid, and then through a bottle "P" containing phosphoric anhydride opened up with glass wool. It then passes at the rate of 100 cc. per minute into the ⅞″x30″ silica tube "T".

Place the boat containing the millings in the silica tube "T", and insert stopper which is connected with a weighed U tube "P", containing phosphoric anhydride opened up with glass wool. This "U" tube is conneceted with a short length of rubber tubing to the glass tube "I", which passes through the small furnace "O" which is maintained at a temperature of 150° C., with the use of a Bunsen burner. This tube contains iodine pentoxide which oxidizes the carbon monoxide to carbon dioxide, the latter being absorbed in a .2 "N" solution of barium hydroxide contained in a Meyer tube "B". The iodine which is formed by the reaction is absorbed by the barium hydroxide, but does not interfere with the precipitation of barium carbonate. The iodine acts upon the rubber tubing making it brittle, this action can be lessened by passing a glass rod greased with vaseline through the new rubber tubing.

After heating for 30 minutes at 1000° C., the gas is turned off and the air blast allowed to cool the silica tube "T", for ten minutes. The absorption tube is detached from the apparatus and is connected with the aspirator shown in engraving. About 500 cc. of air purified by passing through stick potash, "K", sulphuric acid "S", and phosphoric anhydride "P" respectively, is passed through the weighed U tube "X". This is done for the purpose of displacing the hydrogen gas and prevents errors which may arise should the tubes be weighed filled with hydrogen, some of which may be displaced by air should the stopper become dislodged. Another advantage is that the U tubes are quickly cooled by aspirating air through them, so that the errors from weighing tubes at different temperatures are eliminated.

The increased weight of the tube "P", due to the water which was absorbed is multiplied by .8888, divided by the weight taken and multiplied by 100, which gives the per cent of oxygen.

The barium carbonate is filtered on an eleven cm. filter paper, the bulb and paper being washed with boiled distilled water free from carbon dioxide. The filtering should be done at a location where there is no fuel being burned, otherwise some carbonic acid gas would be absorbed. The filter paper containing the barium carbonate is ignited first at low temperature and finally at a red heat, and the white barium carbonate weighed. This figure is multiplied by .1418, divided by the weight taken and multiplied by 100 which gives the per cent of carbon monoxide present.

A blank determination is run on the apparatus each day, the apparatus being adjusted until the final blank on the U tube amounts to less than .003 grams. The final figure is subtracted from the results obtained from each determination. With the use of electrolytic hydrogen there will be no blank to be subtracted from the barium carbonate.

The following results have been obtained on various samples of iron and steel:

Material	Oxygen	Carbon Monoxide
Bureau of Standards, No. 8a	.064	.065
Bureau of Standards, No. 30	.024	.029
American Ingot Iron	.027	.013
Basic Open Hearth Steel	.024	.073
Puddled Iron	.572	.076
Iron Link Newburyport Bridge	.027	.020

J. R. Cain, Bureau of Standards Technologic Paper No. 118, in studying the Ledebur Method for determining Oxygen in iron and steel has developed an electrolytic method for producing pure hydrogen. The following is a description of the method:

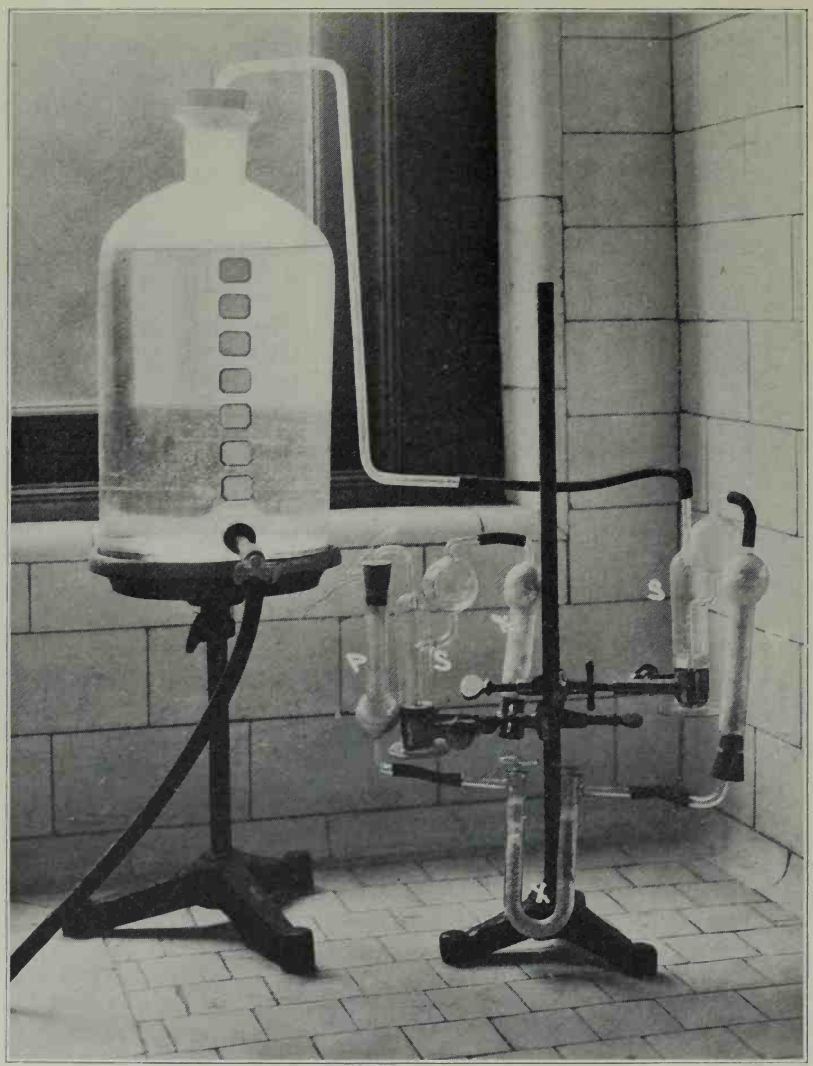
Electrolytic Hydrogen Generator and Reservoir

Hydrogen gas was generated by the electrolysis of a saturated solution of barium hydroxide mixed with a 25 per cent solution of sodium hydroxide in a large pyrex glass U tube, using a platinum anode and a nickel cathode. Platinum and nickel were used as the electrode materials because they have a low oxygen and a low hydrogen overvoltage, respectively.

The generator (Page 160) consists of a U tube containing the electrolyte and submerged in a jar through which flows cold water. The bottom of the U is filled with sea sand to hinder the passage of dissolved gas from one limb of the U to the other, as suggested by Lewis, Brighton, and Sebastian.* The current passing through the generator is regulated by means of a rheostat in the circuit. During the course of the investigation the current used by the operator was 3.3 amperes, which liberates about 1.5 liters of hydrogen gas per hour.

The hydrogen gas reservoir and pressure-maintaining bottle is connected to the cathode side of the generator, and to the anode side a small U tube of 10 mm. inside diameter is attached which contains mercury to balance the pressure in the receiving system. With this arrangement the generator operates automatically. As hydrogen is generated and delivered to the gas reservoir water is displaced from

* J. A. C. S. 39, 1917, 2248



Determination of Oxygen in Iron and Steel

APPARATUS FOR DISPLACING HYDROGEN IN ABSORPTION TUBE WITH DRY AIR

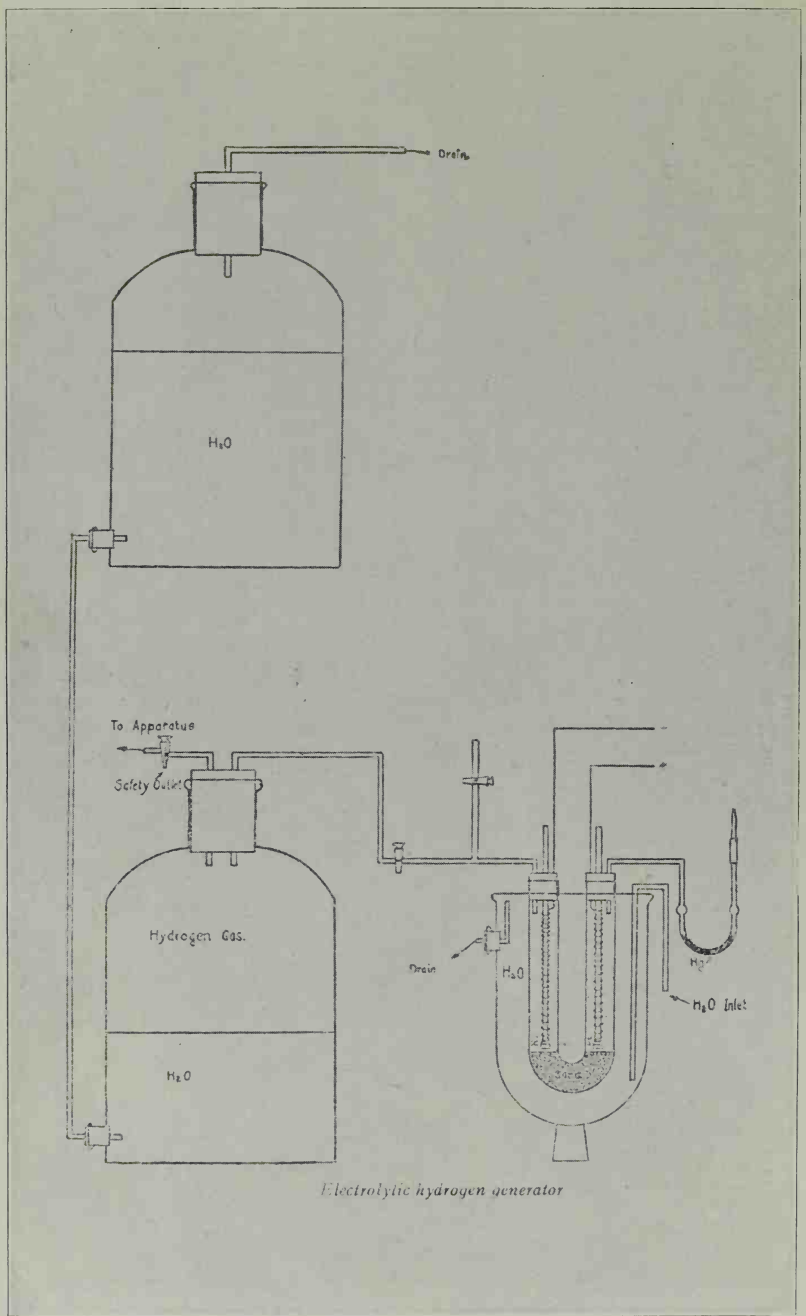
K—Tubes containing calcium chloride.

S—Tubes containing concentrated sulphuric acid.

P—Tubes containing phosphoric anhydride on glass wool.

X—Absorption U Tube, used for weighing the moisture obtained from oxygen in the sample.

the gas reservoir and forced into the water bottle above. The increased pressure thus produced forces down the liquid in the cathode side of the generator an amount approximately equal to the height to which the water level in the water bottle is raised. After a certain volume of hydrogen gas has been generated and stored in the reservoir, the level of the electrolyte on the hydrogen side of the generator will be forced down out of contact with the cathode, thus automatically breaking the circuit. The pressure of the hydrogen that is desired for the experiment is regulated by adjusting the height of the water bottle, which then determines the amount of mercury that must be added to the U tube on the oxygen side of the generator. The hydrogen gas as it is drawn off from the reservoir for use is passed through the catalyzer and purifying train.



Electrolytic hydrogen generator

PHOSPHORUS

DETERMINATION OF PHOSPHORUS
ALKALI TITRATION METHOD

Dissolve 2 grams of the sample in 40 cc. of nitric acid, 1.18 specific gravity, using a 300 cc. Erlenmeyer flask. Heat on hot plate until metal is in solution and add 5 cc. of saturated solution permanganate of potash.

Boil until brown precipitate is formed. Now add four cc. of hydrochloric acid, 1.20 specific gravity or a sufficient amount to clear the solution by boiling a few minutes. Avoid an excess of hydrochloric acid as it interferes with the precipitation of phosphorus when extremely low.

Remove from hot plate, cool somewhat, and cautiously add ammonia, .90 specific gravity, shaking flask occasionally, until a heavy precipitate of ferric hydroxide is formed. Then add nitric acid, 1.42 specific gravity, shaking occasionally, until precipitate dissolves and a clear amber-colored solution is obtained.

It is very essential that an excess of nitric acid should be avoided, as it interferes with the precipitation of phosphorus when this element exists in traces.

Heat or cool solution to 85° C., and add 50 cc. of ammonium molybdate solution. Shake well and allow to stand at least ½ hour or until precipitate settles.

Filter and wash with 2% nitric acid solution until free from iron, and finally with distilled water containing about 1 gram of potassium nitrate to liter until free from acid.

Transfer filter and contents to tumbler containing 50 cc. of boiled distilled water. Disintegrate paper with two stirring rods and add sufficient standard sodium hydroxide to dissolve the yellow precipitate and render the solution pink when phenolphtalein indicator is added. Now run in standard nitric acid until pink color disappears, then finish the titration with standard alkali, the end point being a faint pink color.

TITRATION EXAMPLE

Standard Alkali		Standard Acid	
Last Reading	27.5 cc.	Last Reading	14.7 cc.
First Reading	17.3	First Reading	6.7
	10.2 cc.		8.0 cc.

2.2 cc. x .01 = .022% Phos.

Standard Solutions

The standard nitric acid and alkali used for titrating are about .15 normal. One cc. being equal to .01% Phosphorus when a 2-gram sample is used for analysis.

A stock solution of sodium hydroxide is prepared by dissolving 192 grams of sodium hydroxide in water and adding enough barium hydroxide solution to precipitate all carbonates, then diluting to two liters. Use 50 cc. of stock solution diluted to two liters for the standard alkali solution.

A stock solution of nitric acid for titrating can be prepared by mixing 367 cc. of concentrated nitric acid, specific gravity 1.42, with enough boiled distilled water free from carbon dioxide to make two liters. Use 50 cc. stock solution diluted to two liters for the standard acid solution.

Nitric Acid for dissolving the sample can be prepared by adding 1 part of nitric acid, 1.42 specific gravity, to 2 parts of water. The specific gravity of this mixture will be very close to 1.18.

Ammonium Molybdate Solution

Preparation

Place in 5-pint bottle:
500 cc. Conc. HNO_3, Sp. Gr. 1.42.
1700 cc. Distilled Water.
Place in 400 cc. beaker:
90 g. Molybdic Acid.
100 cc. Distilled Water.
100 cc. Conc. Ammonia.

Add ammonium molybdate slowly to acid in bottle while stirring. Mix thoroughly then *add 2 drops only* saturated ammonium phosphate solution. Agitate and let settle.

Use 40 cc. to 50 cc. of clear solution.

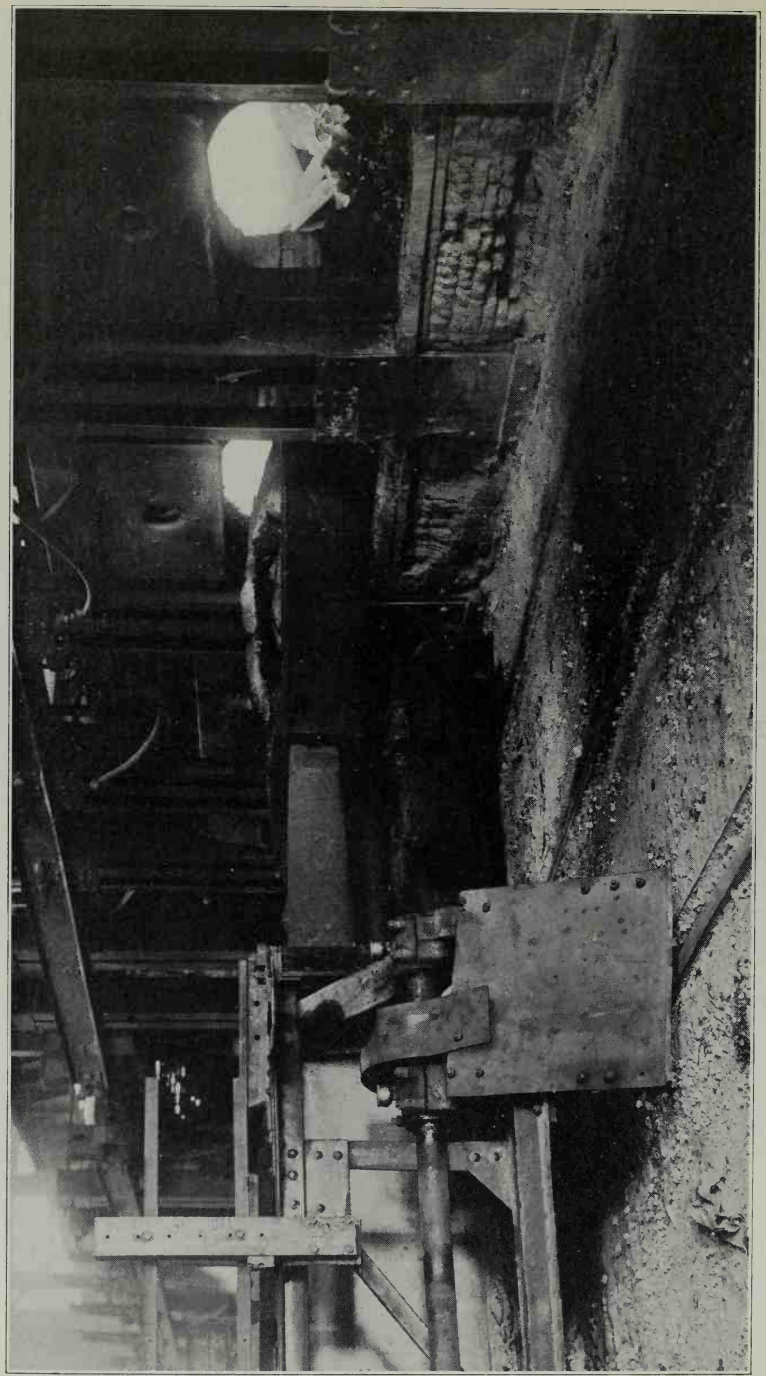
MODIFICATION FOR DETERMINING PHOSPHORUS IN CHROME-VANADIUM STEEL

On account of the vanadium interfering with the determination of phosphorus we use the method of Hagmaier, described in Metallurgical and Chemical Engineering, Vol. XI, p. 28. This method is about as follows:

Dissolve 2 g. of the steel in aqua regia in a 4-in. casserole, evaporate to dryness and bake. Cool, dissolve in 35 cc. of concentrated hydrochloric acid, dilute with water and filter from silica.

Reduce the filtrate with sulphurous acid. When entirely reduced add 5 cc. of 90% acetic acid and 10 cc. of a saturated solution of cerium chloride. Add dilute ammonia with constant stirring until the solution becomes turbid. Then heat the solution to boiling, allow to settle, and filter. The cerium phosphate will filter rapidly. Wash the precipitate several times with hot water and then dissolve off the paper with hot 1:1 nitric acid.

Precipitate the phosphorus from this solution in the regular manner with ammonium molybdate and titrate with alkali as previously described. Add ammonia very slowly, as it is impossible to obtain proper conditions if an excess is added and an attempt is made to neutralize with acid. Should an excess inadvertently be added it is best to start another determination instead of attempting to neutralize the excess of ammonia.



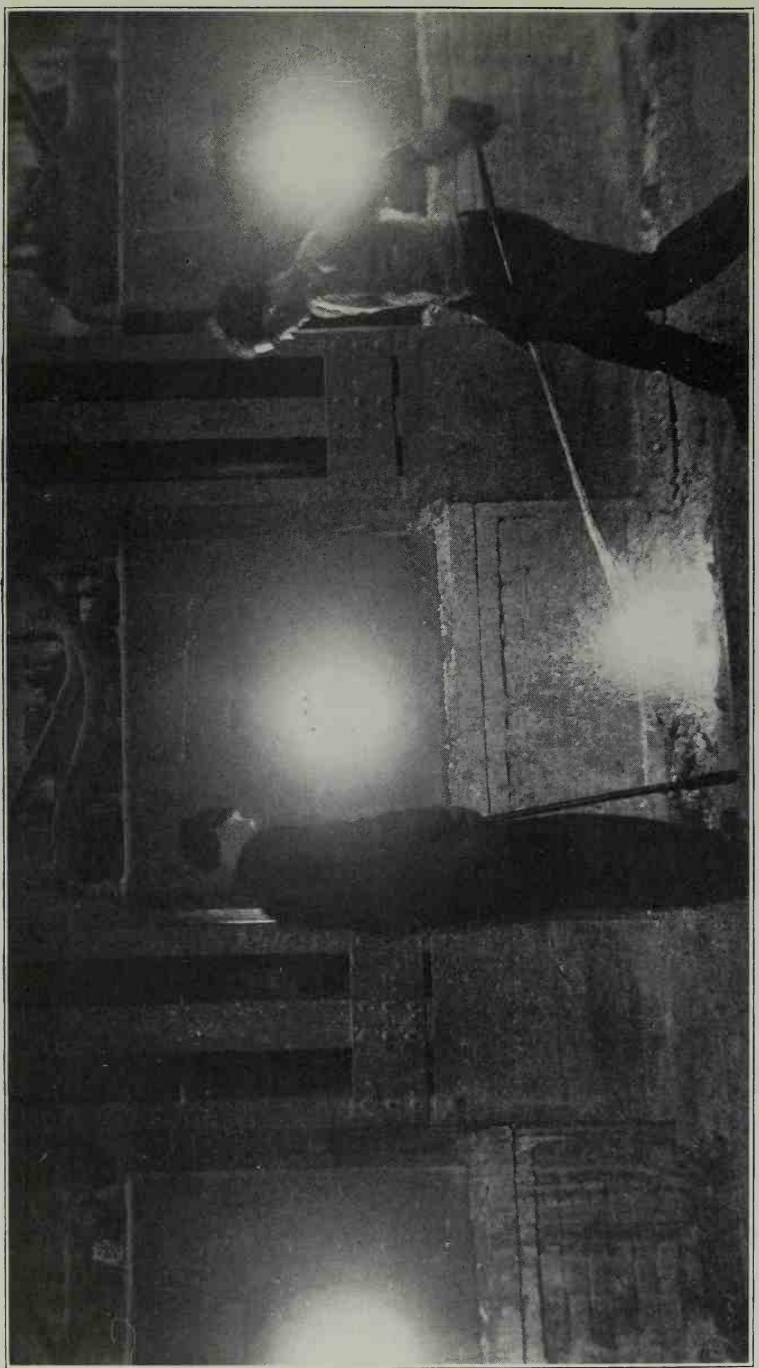
View of 125 ton Basic Open Hearth Furnace showing the hot pig iron in the furnace and a pan of pig iron ready to be dumped into the furnace by the charging machine

PIN HOLE TEST

LEAD COATED, TIN AND TERNE PLATE

Dr. Allerton S. Cushman has devised a very simple test to determine the number of pin holes per square foot. The test consists of exposing a full sized sheet to the action of distilled water. The pin holes appear as rust spots.

The four sides of the sheet are bent so as to make a pan 1″ deep. The pan is thoroughly cleaned with several applications of gasoline and then flooded to a depth of ½″ with distilled water. After one week's exposure the water is removed and the pin holes are counted.



Close view of 125-Ton Basic Open Hearth Furnace, showing three port holes from which the glare of the hot metal can be seen. This picture illustrates the method of taking a test for chemical analysis. The spoon is placed in the furnace and filled with molten metal which is poured into a cast iron mold, the sample thus obtained is taken to the laboratory. No heat is tapped until the chemical analysis shows the product to be within specifications

SILICON

THE DETERMINATION OF SILICON IN IRON AND STEEL

Dissolve 4.69 grams of the sample in a platinum dish, using 60 cc. of nitric acid, 1.18 specific gravity, and 10 cc. of sulphuric acid, 1.84 specific gravity. Evaporate to dense white fumes and when cool dissolve ferric sulphate in about 35 cc. of hydrochloric acid, 1.20 specific gravity. Dilute with water and filter through an ashless paper. Wash alternately with distilled water and dilute hydrochloric acid, 1.05 specific gravity, until free from iron.

Ignite in platinum crucible, using a muffle for this purpose. If muffle is not available use a Meker burner with natural draft.

Weigh residue and add about 1 cc. of hydrofluoric acid and about 3 drops of concentrated sulphuric acid. Heat crucible carefully from the top of the crucible instead of from the bottom, and when all acid has evaporated heat to the full temperature of burner until iron has changed to oxide. Cool and weigh. The loss is silica. Each milligram equals .01% of silicon.

Notes on Method

If silicon is determined in alloys containing chromium, the evaporation of the acids should not be done at high temperatures. This evaporation should be conducted on hot plate, as some chromic oxide is liable to separate.

If the silica after ignition is canary yellow in color, it indicates the presence of tungsten. The residue remaining in the crucible after treatment with hydrofluoric acid will consist essentially of tungsten trioxide (WO_3). For accurate results, however, the regular method should be employed for the determination of this element.

If silicon is to be determined in Pig Iron, use 2.35 grams. Dissolve in a porcelain dish instead of a platinum dish, using 50 cc. of the following mixture:

Water.....68%
Conc. Nitric Acid.....23½%
Conc. Sulphuric Acid.....8½%

If silicon is being determined in alloy steels, omit the use of platinum dish and use the following mixture of acids which will prevent bumping or spattering:

Water	55%
Conc. Nitric Acid	25%
Conc. Sulphuric Acid	10%
Conc. Hydrochloric Acid	10%

Note—In the case of Pure Iron and Steel which contains a trace of silicon we use 4.59 grams of the sample, using nitric and sulphuric acid in a platinum dish.

For steel containing appreciable amounts of silicon we use 2.35 grams of the sample and 50 cc. of the above acid mixture in a porcelain dish.

THE DETERMINATION OF SILICON, ALUMINUM, TITANIUM AND ZIRCONIUM IN STEEL

BUREAU OF STANDARDS

Dissolve 5.00 grams of the steel in 50 cc. of hydrochloric acid (sp. gr. 1.2) by gentle warming and the addition of 1 cc. portions of nitric acid from time to time to insure solution of the zirconium and titanium and also oxidation of the iron.

When solution is complete, evaporate to dryness, take up in 10 cc. of hydrochloric acid (sp. gr. 1.2), again evaporate to dryness, and finally bake at a gentle heat in order to decompose nitrates.

Cool, take up in 50 cc. of 1:1 hydrochloric acid, and filter when the iron is completely in solution. Wash the residue with cold 1:1 hydrochloric acid. Save the filtrate and washings.

Ignite the residue and paper in a platinum crucible, cool and weigh. Treat with 1 cc. of sulphuric acid (1:1) and sufficient hydrofluoric acid, fume off in the usual manner, ignite and weigh to obtain silica, and calculate silicon.

Fuse the slight residue left after the hydrofluoric acid treatment with a small amount of potassium pyrosulphate, dissolve in 10-20 cc. of 5% sulphuric acid and add the solution to the acid extract from the ether separation obtained as described below.

Evaporate the filtrate and washings from the silica determination to a syrupy consistency, take up in 40 cc. of hydrochloric acid (sp. gr. 1.1) and extract with ether in the usual manner. (The *ether* extract will contain most of the molybdenum, and this element may be qualitatively tested for in it. If molybdenum is present it is more conveniently determined in a separate portion of steel). The acid extract will contain some iron, and all of the zirconium, titanium, aluminum, nickel, chromium, etc.

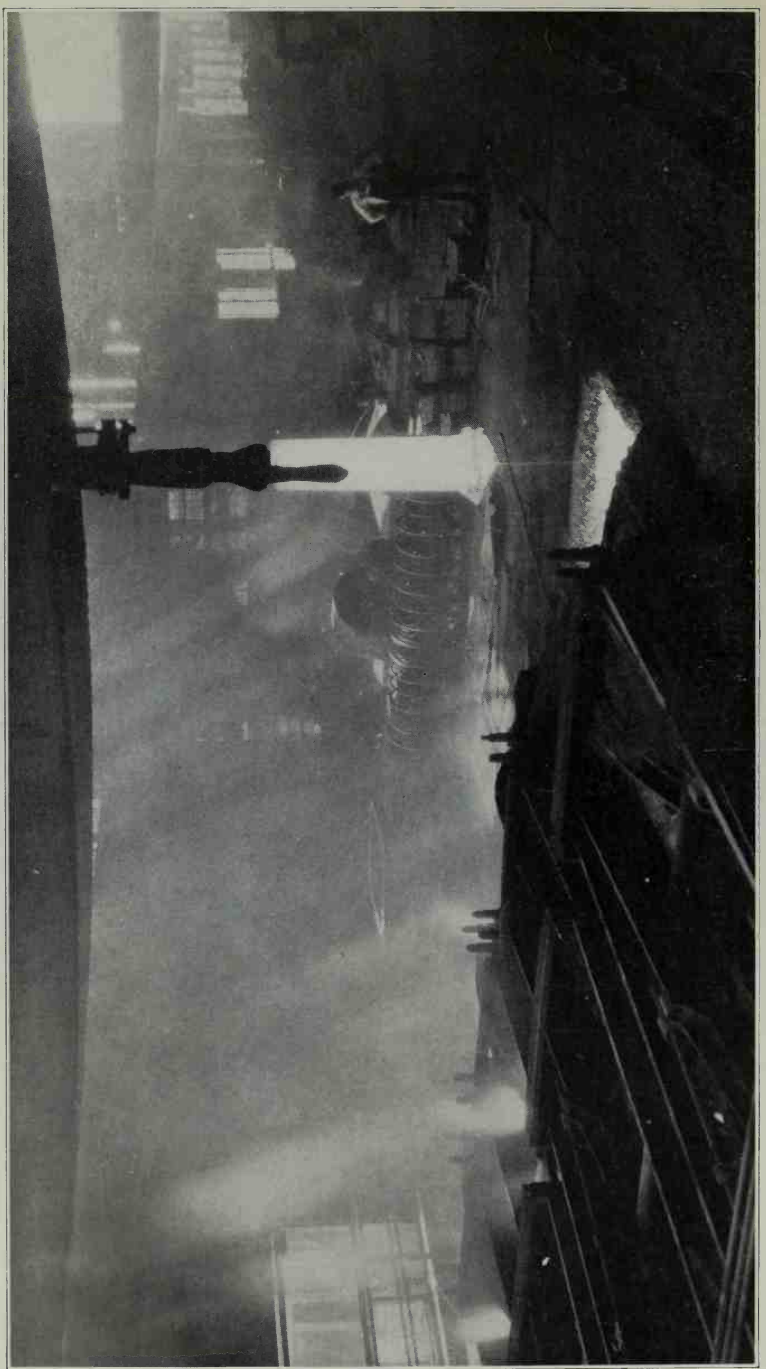
Gently boil off the ether in the acid extract, add the matter recovered from the silica, oxidize ferrous iron with a little nitric acid, dilute to 300 cc, cool and precipitate with 20% sodium hydroxide solution, adding 10 cc. in excess. Filter, and save the filtrate. Dissolve the precipitate in warm dilute 1:1 hydrochloric acid and repeat the precipitation. Combine the sodium hydroxide filtrates. Dissolve the precipitate as above and reserve the solution for subsequent analysis.

It is advisable to treat as follows the filter or filters used above: Ignite in platinum, fuse with sodium carbonate, digest the cooled melt with hot water, wash the residue, discard the filtrate and washings, dissolve the residue in hot 1:1 hydrochloric acid and add to the main acid solution. This precaution makes certain the recovery of any zirconium held back on the filter as zirconium phosphate insoluble in acid.

Five samples of pig iron are taken from each carload received. Samples are broken with a sledge and taken to the chemical laboratory for analysis

DETERMINATION OF ALUMINUM IN THE ABSENCE OF CHROMIUM

Add a few drops of methyl red to the sodium hydroxide, filtrate, neutralize with hydrochloric acid, add 4 cc. of concentrated hydrochloric acid per 100 cc. of solution, boil, make barely alkaline with ammonium hydroxide, continue the boiling for three minutes and then set the beaker aside for ten minutes. If no precipitate settles out, the absence of aluminum is assured. If a white precipitate settles out, aluminum is indicated; this precipitate is always contaminated by phosphorus pentoxide and must be purified as follows: filter without washing, discard the filtrate and dissolve the precipitate in warm 1:1 hydrochloric acid. Dilute the solution to 50 cc., make alkaline with ammonium hydroxide, neutralize with nitric acid and add 2 cc. in excess. Warm to 50° C., precipitate the phosphoric acid with molybdate reagent in the usual manner, filter, and wash the phosphomolybdate with ammonium acid sulphate solution. Precipitate the aluminum in the filtrate as directed above, filter without washing, dissolve the precipitate in warm 1:1 hydrochloric acid, reprecipitate, filter, wash slightly with 2% ammonium chloride solution and ignite in a platinum crucible. The ignited residue is usually contaminated by silica, therefore a sulphuric acid-hydrofluoric acid treatment followed by ignition over the blast lamp to alumina should be performed. (The sodium hydroxide reagent must be tested for substances which are precipitated by ammonia, and appropriate corrections must be made in the aluminum determination when these are present).



White hot ingot taken from soaking pit ready to be transferred on a live roll table to the Blooming Mill

DETERMINATION OF ALUMINUM IN STEELS CONTAINING CHROMIUM

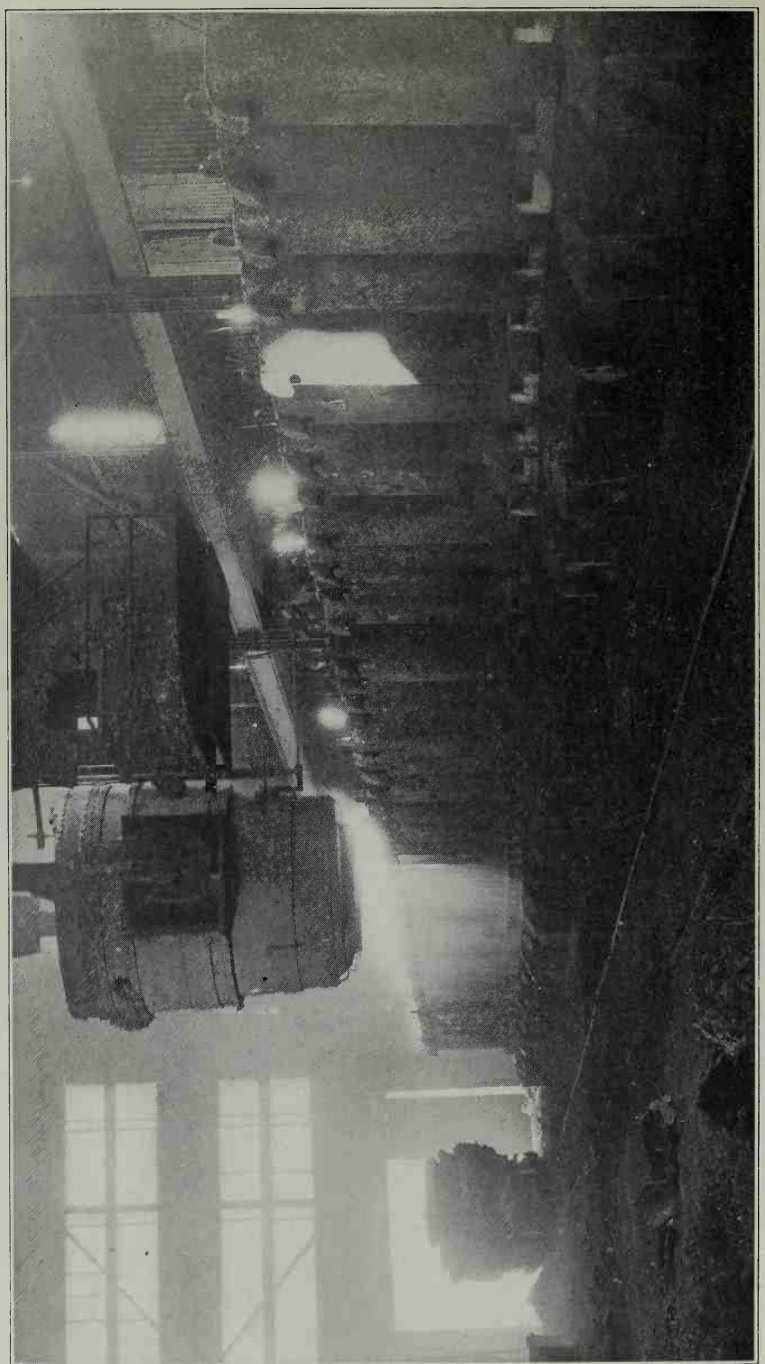
Proceed as above until the filtrate from the molybdate precipitation is obtained. Then make the solution ammoniacal, oxidize with a little bromine water, make just acid with 1:2 nitric acid, add ammonium hydroxide in slight excess, heat to boiling, filter, dissolve the precipitate in dilute hydrochloric acid, and reprecipitate the aluminum hydroxide as directed above.

DETERMINATION OF ALUMINUM IN STEELS CONTAINING URANIUM

The only modification which is required is the substitution of ammonium carbonate for ammonium hydroxide as the final precipitant of the aluminum hydroxide.

DETERMINATION OF ALUMINUM IN STEELS CONTAINING VANADIUM

Alumina which is obtained by the above procedures, from steels containing vanadium, is contaminated by this element. When dealing with these steels proceed as follows: fuse the weighed residue with pyrosulphate, extract the cooled melt with 5% sulphuric acid, reduce the vanadium in a Jones reductor having ferric alum in the receiver, titrate the reduced solution with standard permanganate, calculate the vanadium as V_2O_5 and subtract from the original weight.



Ladle containing 125 tons of molten iron which is being poured from a nozzle in the bottom of the ladle into the ingot molds on buggies. These buggies are moved to the soaking pits where the molds are stripped from the ingots. The ingots are then placed in gas heated pits and heated uniformly for rolling

DETERMINATION OF ZIRCONIUM AND TITANIUM

Dilute the hydrochloric acid solution to 250 cc., neutralize with ammonium hydroxide so as to leave approximately 5% (by volume) of hydrochloric acid, add 2 grams of tartaric acid, and treat with hydrogen sulphide until the iron has been reduced. Filter if the sulphide group is indicated. Make the hydrogen sulphide solution ammoniacal and continue the addition of the gas for 5 minutes. Filter carefully and wash with dilute ammonium sulphide-ammonium chloride solution. Filter through a new filter if the presence of iron sulphide in the filtrate is indicated. Save the filtrate.

(The sulphide precipitate consists of ferrous sulphide, in addition to the greater part of any nickel, cobalt and manganese present in steel. It is preferable to determine these in separate portions of the steel).

Neutralize the ammonium sulphide filtrate with sulphuric acid, add 30 cc. in excess and dilute with water to 300 cc. Digest on the steam bath until sulphur and sulphides have coagulated, filter, wash with 100 cc. of 10% sulphuric acid and cool the filtrate in ice water.

Add slowly and with stirring an excess of a cold 6% water solution of cupferron. (The presence of an excess is shown by the appearance of a white cloud which disappears, instead of a permanent coagulated precipitate). Immediately filter on paper, using a cone and very gentle suction, and wash thoroughly with cold 10% hydrochloric acid.

Carefully ignite in a tared platinum crucible, completing the ignition over a blast lamp or large Meker, cool and weigh the combined zirconium and titanium oxides.

Fuse with potassium pyrosulphate, dissolve in 50 cc. of 10% (by volume) sulphuric acid and determine titanium colorimetrically or volumetrically. Calculate titanium oxide and subtract the weight found from that of the combined oxides and calculate zirconium.

Notes:

1. Phosphorus pentoxide contaminates the precipitate to so slight an extent that it can be disregarded.

2. Vanadium interferes no matter what its valency. The interference is not quantitative. If present in the steel, proceed as usual through the weighing of the cupferron precipitate. Then fuse thoroughly with sodium carbonate, cool, extract with water, filter,

and determine the vanadium in the filtrate by adding sulphuric acid, reducing through a Jones' reductor into a solution of ferric phosphate and then titrating with standard permanganate. Ignite in the original crucible the matter insoluble in water, fuse with potassium pyrosulphate and proceed as directed for titanium.

3. Tungsten does not interfere since it is separated from zirconium and titanium by the sodium hydroxide treatment, and from aluminum by the ammonium hydroxide precipitation. If tungsten is present in large amount it may be found desirable to fuse the nonvolatile residue from the silicon determination with sodium carbonate, extract with water, filter, dissolve the residue in hot 1:1 hydrochloric acid and add to the acid extract from the ether separation.

4. Uranium is partially carried down when present in the tetravalent condition, but at not all in the hexavalent state. If this element is suspected, boil out all hydrogen sulphide before the cupferron precipitation, oxidize with permanganate to a faint pink, cool and proceed with the cupferron precipitation.

5. Thorium and cerium interfere, but they are not thrown down quantitatively. In case these elements are suspected, the peroxidized solution used for the titanium determination must be quantitatively preserved and reduced with a little sulphuric acid. The rare earths are then separated by Hillebrand's method[1] as follows: precipitate the hydroxides with an excess of potassium hydroxide, decant the liquid, wash by decantation with water once or twice and then slightly on the filter. Wash the precipitate from the paper into a small platinum dish, treat with hydrofluoric acid, and evaporate nearly to dryness. Take up in 5 cc. of 5% (by volume) hydrofluoric acid. If no precipitate is visible, rare earths are absent. If a precipitate is present, collect it on a small filter held by a perforated platinum or rubber cone and wash it with from 5 to 10 cc. of the same acid. Wash the crude rare-earth fluorides into a small platinum dish, burn the paper in platinum, add the ash to the fluorides and evaporate to dryness with a little sulphuric acid. Dissolve the sulphates in dilute hydrochloric acid, precipitate the rare-earth hydroxides by ammonia, filter, redissolve in hydrochloric acid, evaporate the solution to dryness, and treat the residue with 5 cc. of boiling hot 5% oxalic acid. Filter after fifteen minutes, collect the oxalates on a small filter, wash with not more than 20 cc. of cold 5% oxalic acid, ignite and weigh as rare-earth oxides which are to be deducted from the weight of the cupferron precipitate.

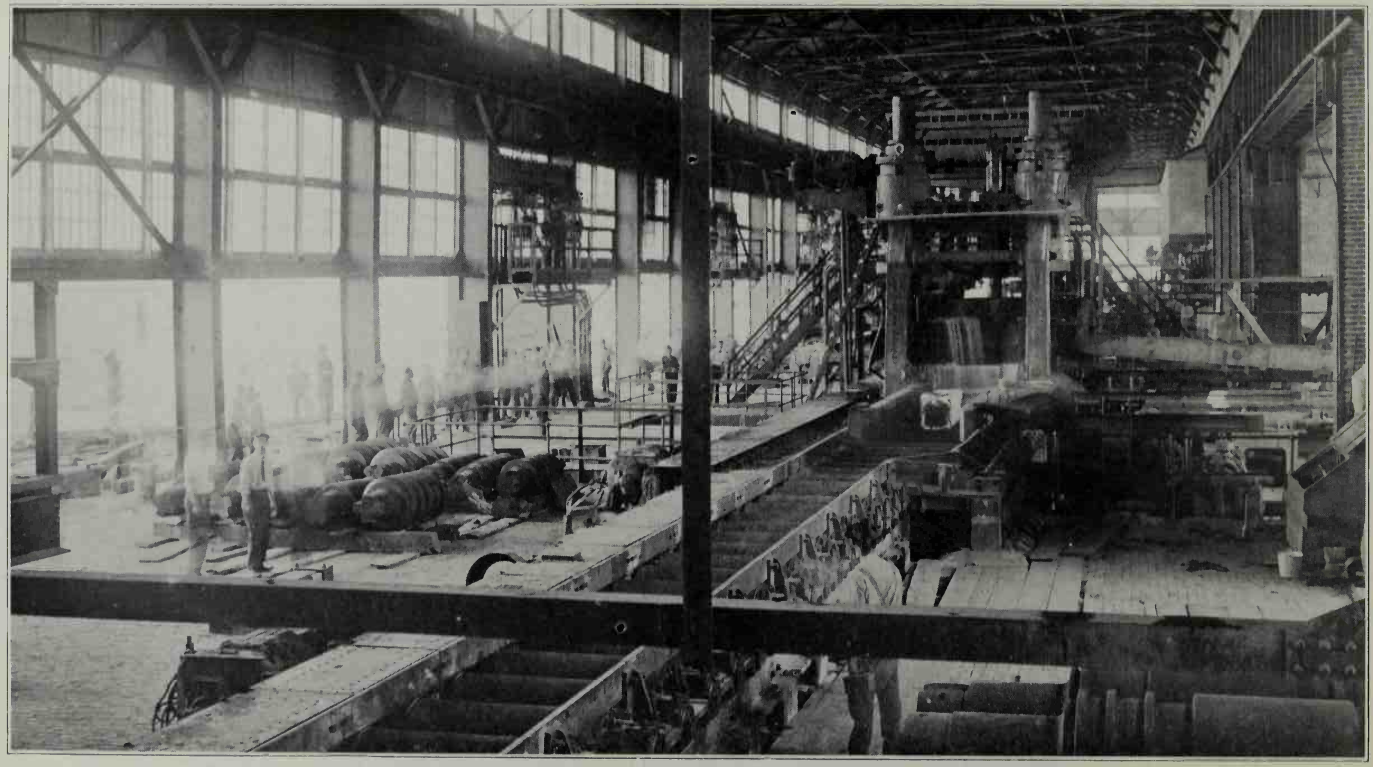
1 U. S. Geol. Survey Bulletin 700. The Analysis of Silicate and Carbonate Rocks, p. 176.

The foregoing procedure does not give an absolutely quantitative recovery of the rare earths. Experiments indicate a recovery of approximately 85% of the rare earths present in residues containing 100 mg. of zirconia, 2 mg. of thoria and 2 mg. of ceria.

Attempts which were made to omit the preliminary separation of the rare earths as fluorides were unsuccessful.

6. Instead of the prescribed treatment for the removal of the bulk of the iron, Johnson's[2] method of fractional ammonium hydroxide precipitation may be used. When using this method, it is recommended that the 1:1 hydrochloric acid solution of the ammonium hydroxide precipitate should be further treated as given in the Bureau of Standards method beginning with "oxidize. . . . . .and precipitate with a 20% sodium hydroxide solution". In Johnson's procedure silicon must be determined in a separate portion.

2 C. M. Johnson, Chem. and Met. Eng. 20, 1919, p. 588.



View of ingot about to enter the Blooming Mill rolls. Water is allowed to flow onto these rolls in order to keep them cool. By successive passes through this mill ingots are drawn out to slabs or bars of a size suitable for finishing in the bar mill.

CUSHMAN'S METHOD FOR THE DETERMINATION OF SPELTER COATING BY MEASURING THE HYDROGEN EVOLVED[1]

The determination of the weight of spelter coating by this method is based upon the action of hydrochloric acid upon the galvanized coating, collecting and measuring the hydrogen gas evolved[2]. The weight of coating may be determined upon flat sheets, corrugated sheets, and formed culverts, by the use of differently shaped rings provided with the apparatus. The coating upon wire can be determined by placing a definite length of wire under the flat ring on a glass plate.

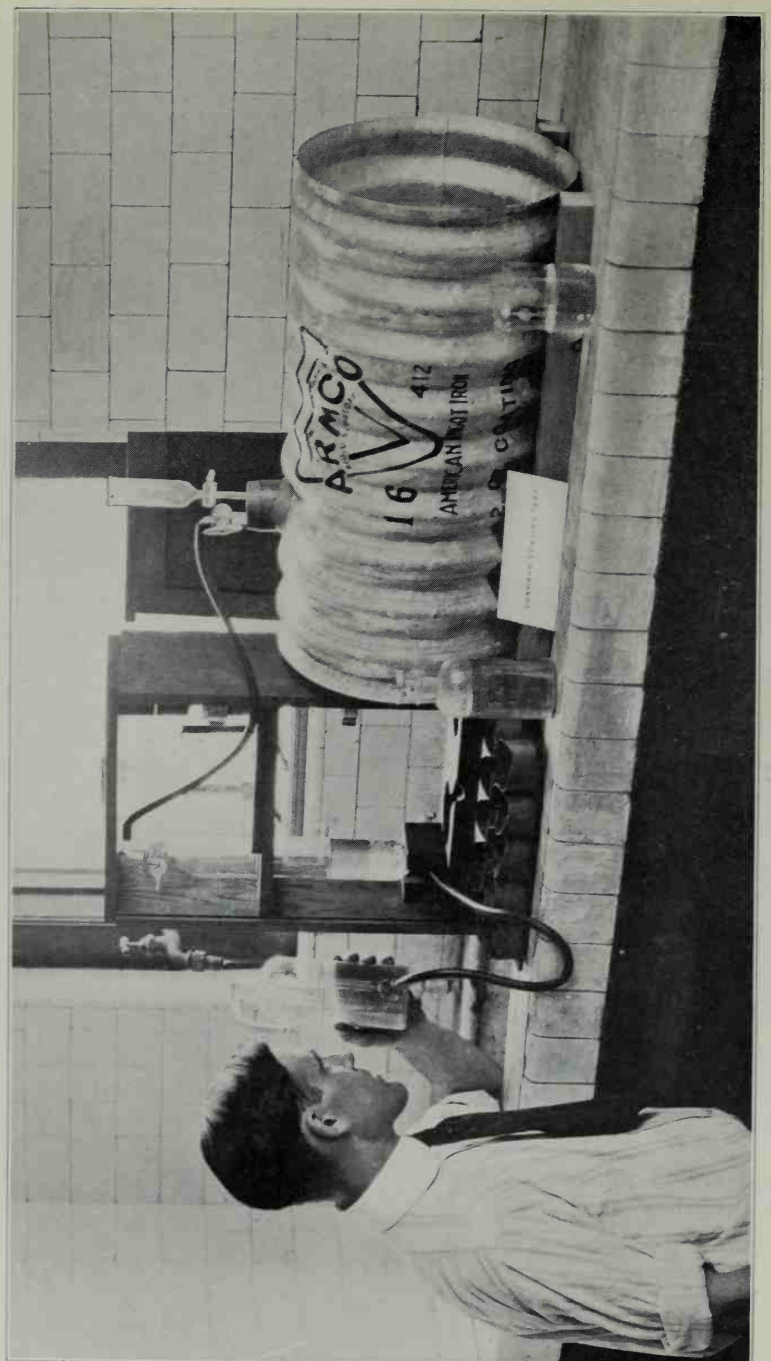
The metallic rings are made of nickel, tinned iron, or other acid resisting metal, and are fitted with No. 12, three-hole rubber stoppers. Through one hole passes the filling tube provided with glass stopcock. Through the other holes pass the exit tubes, the short tube to a position even with the bottom of the stopper, the long tube extending to a position even with the bottom of the ring. A measuring burette and leveling bottle are provided for collecting and measuring the hydrogen evolved.

The measuring burette is first filled with water, allowing a small amount of water in the leveling bottle. The proper ring is selected for the culvert to be tested, and is placed upon the culvert and sealed with "Plasteline", or other acid resisting wax. The stopcock on the acid tube is turned so as to communicate with the short tube, and is then connected with the measuring burette by means of a rubber tube. Water is now placed in the filling tube, the stopcock opened, and the ring and connecting tubes completely filled with water by lowering the leveling bottle, and allowing the air to flow into the burette. By means of the three way stopcock on the measuring burette, it is again filled with water without disturbing the water in the ring.

The stopcocks in the measuring burette are opened and the stopcock on the exit tube turned to connect the long tube with the burette. If there are any leaks in the apparatus the water in the measuring burette will fall. With everything prepared and ready, about 30 cc. of concentrated hydrochloric acid are placed in the filling tube and about 5 cc. admitted to the ring. The hydrogen generated from the zinc will force out the water in the ring. As soon as gas appears in the long exit tube, the stopcock is quickly reversed to the

1 Dr. A. S. Cushman, Proceedings of The American Society for Testing Materials, 1920.

2 The apparatus for making this test can be obtained from the Kauffman-Lattimer Co., Columbus, Ohio.



Determination of the weight of Spelter Coating by the Cushman Method

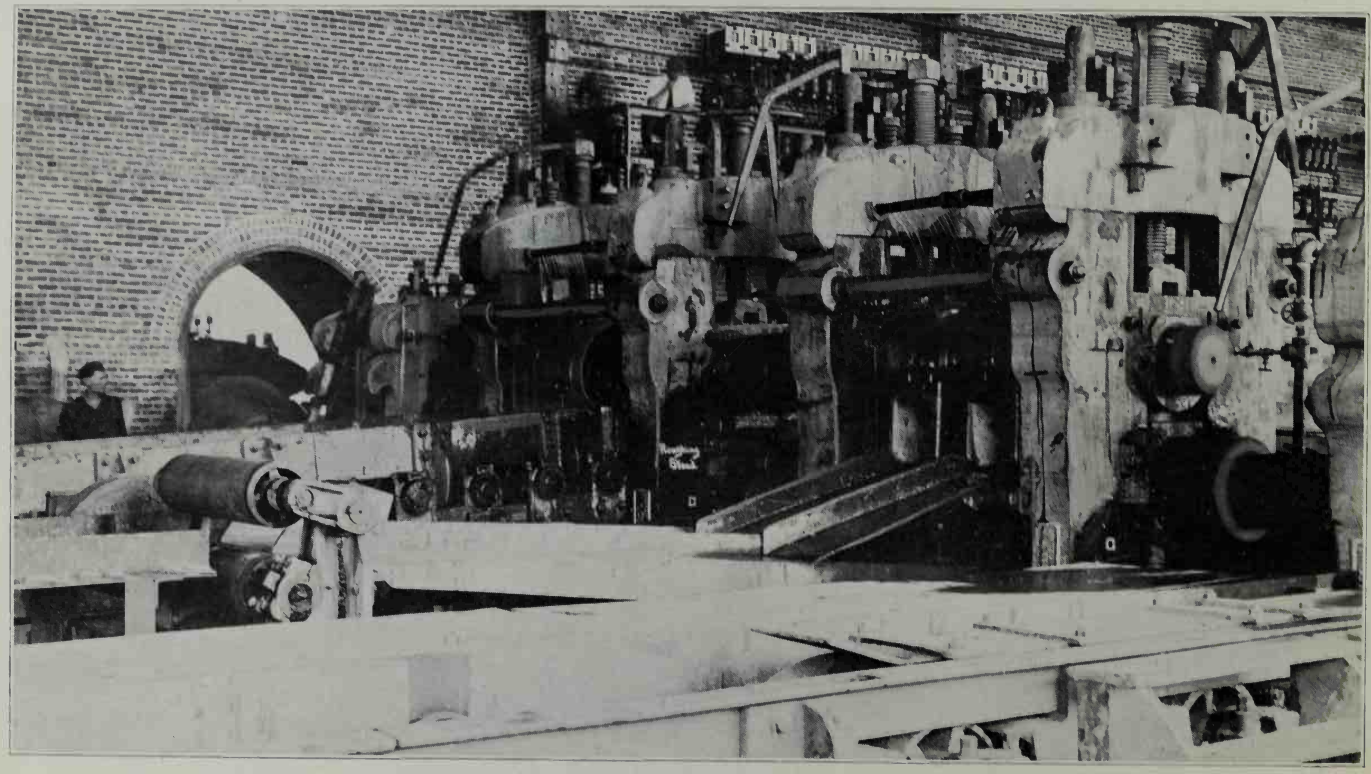
short exit tube, 3 cc. of antimony chloride solution[2] are added to the acid in the filling tube and the acid allowed to run into the ring.

When the generation of gas has ceased, the ring and connecting tube are completely filled with water through the filling tube by lowering the leveling bottle, and as soon as the liquid reaches the burette the stopcock is turned off, the water in the leveling bottle and burette brought to the same level and the volume of hydrogen recorded.

The burette stopcock is now turned to communicate with the waste beaker and enough water passed through the ring by means of the filling tube to remove all acid. By turning the stopcock to the long exit tube the ring can be completely drained. In case the ring is lower than the burette stopcock it is necessary to blow out the water with a rubber tube and stopper inserted in the filling tube. The ring can then be removed and the spot on the culvert cleaned with gasoline. The spot may then be coated with a paste of zinc powder and zinc chloride (50% solution) and heated with a blowtorch until fused, or it may be coated with a zinc powder paint or aluminum paint.

The number of cubic centimeters of hydrogen measured at 20° C. (75° F.) multiplied by the factor provided with each ring will give the ounces of spelter coating per square foot of actual surface on one side of the culvert. By doubling this figure the coating in ounces per square foot of sheet surface can be obtained. The factors for each ring are given in a table accompanying the apparatus.

2 Five grams of antimony chloride dissolved in 100 cc. of concentrated hydrochloric acid, Sp. Gr. 1.20.



After the ingots have been rolled into slabs on the Blooming Mill, the material is cropped until all imperfect metal is discarded. The remainder is cut into several sections which are rolled on the bar mill shown in this picture. These bars range in thickness from $\frac{1}{2}$" to $1\frac{1}{2}$" depending upon the length and thickness desired in the finished sheet.

THE DETERMINATION OF SPELTER COATING ON SHEETS AND WIRE

HYDROCHLORIC ACID METHOD

For many years the Preece copper-sulfate test has been used to determine the amount of galvanized coating on sheets and wire. Committee A-5 on the Corrosion of Iron and Steel, The American Society for Testing Materials, reported to the Society in 1911 on this test as follows:

"It is, however, the unanimous opinion of the committee that the well-known Preece copper-sulfate test is unreliable and should be abandoned entirely as a basis of specification with respect to galvanized sheet and plate. In respect to wire, the Preece test has the advantage of being quick and simple, and if carried out in the proper manner, yields comparative results of value. In the opinion of the committee, the lead-acetate is preferable to the copper-sulfate test for determining or specifying the weight of zinc coatings."

The lead-acetate method recommended by Committee A-5 in 1911 yields very accurate and satisfactory results, but the length of time required for making the test seriously limits the scope of its usefulness. The results obtained with the method described in this paper compare very favorably with those of the lead-acetate method.

There is much to be desired in the method of expressing the weight of coating on wire products in order to have an intelligent understanding as to the weight of coating per unit area. It has been customary to express the weight of coating on wire in pounds per mile, while on sheet products the results are usually expressed in ounces per square foot. Obviously, the coating on wire expressed in pounds per mile would have a different meaning for each gage of wire. If the results are expressed in ounces per square foot of surface on both wire and sheets, there will be a better understanding as to the thickness of coating on the respective products. In stating the weight of coating on galvanized sheets it is customary to express the weight based on one surface only, that is, a sheet containing 2 oz. of coating per square foot really contains 1 oz. on each side of the sheet.

It is proposed to express the weight of coating on wire in ounces per square foot, and also to use such lengths of wire that the number of grams of coating found will be equivalent to ounces per square foot,

without calculation. These lengths must be such that the surface coated is equal to 5.079 sq. in. It is likewise proposed that the samples for determining the weight of coating on galvanized sheets shall be 2¼ by 2¼ in. (area = 5.079 sq. in.). The number of grams of coating on a section of this size will also express the weight of coating in ounces per square foot without calculation.

The method for determining the weight of spelter coating consists of using a small amount of antimony chloride in hydrochloric acid (sp. gr. 1.20). Antimony chloride appears to hasten the solution of the coating, and after the coating has dissolved a thin film of antimony plates on the surface of the base metal and retards the solution of iron or steel. Experiments have shown that sheet steel 2¼ by 2¼ in. which loses 50 mg. in five minutes in cold hydrochloric acid (sp. gr. 1.20), will lose in that time only 1 mg. in the same acid containing 80 mg. of antimony per 105 cc. of acid.

In the proposed method the metal is immersed in the acid only one minute, which is long enough to dissolve several grams of coating, yet the amount of iron or steel dissolved is negligible. The small amount of antimony that plates on the surface of the sample can easily be removed by scrubbing under running water. This method is one of the most rapid and accurate with which the writer is familiar, and a determination can be made in less time than is occupied in making the Preece test.

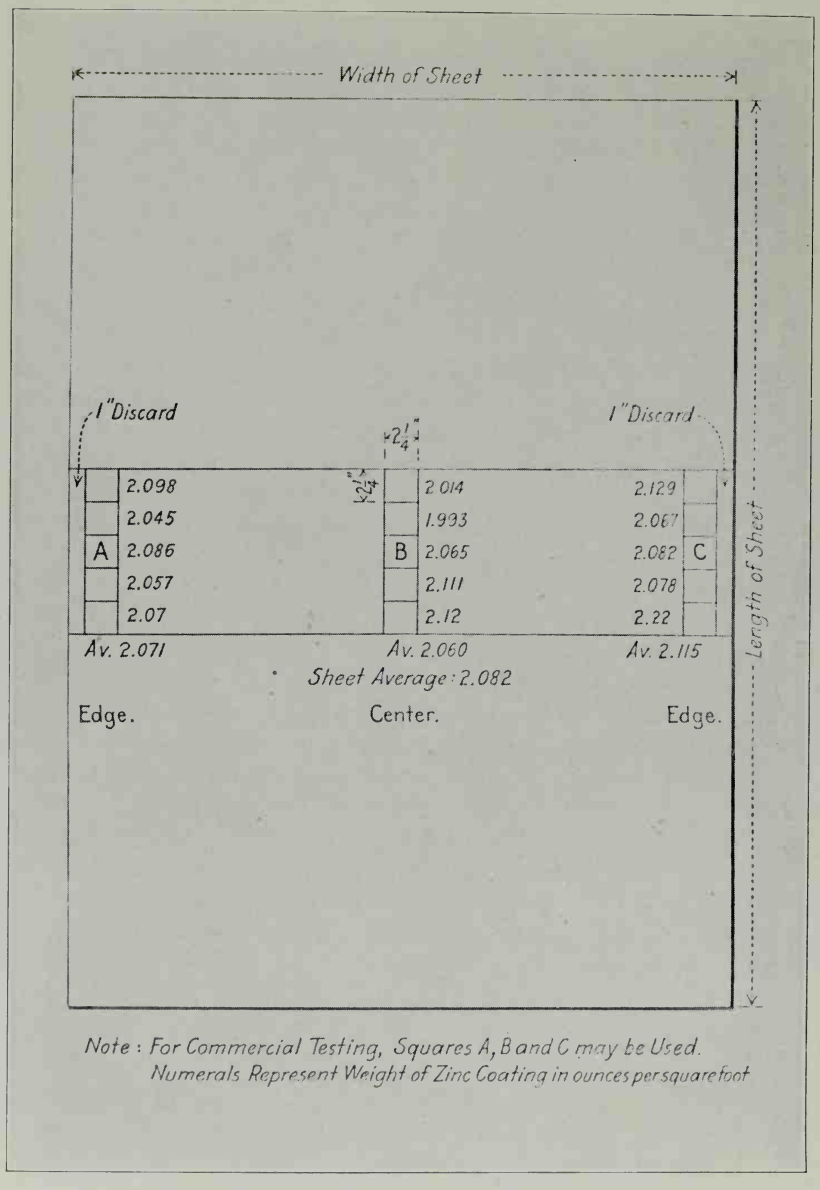
SHEETS—For determining the weight of coating on galvanized sheets, cut three samples 2¼ by 2¼ in. from a strip cut from middle of sheet as shown on Page 186. The three samples should be weighed together and immersed singly for one minute in 100 cc. of hydrochloric acid (sp. gr. 1.20), to which has been added 5 cc. of antimony chloride prepared by dissolving 20 g. of antimony trioxide in 1000 cc. of hydrochloric acid (sp. gr. 1.20). The same 100 cc. of hydrochloric acid can be used for at least five samples. Five cubic centimeters of the antimony chloride, however, should be added for each sample on account of the antimony being removed from the solution by the iron.

The samples are washed and scrubbed under running water, dried with a towel, and laid in a warm place for a few seconds. The samples are again weighed together and the number of grams lost is divided by the number of samples taken. Each gram corresponds to 1 oz. of coating per square foot.

Wire—A small section of the galvanized wire should be stripped in hydrochloric acid containing antimony chloride. The diameter of the black wire should then be carefully measured in order to determine the length of wire, such that the number of grams of coating will represent the number of ounces per square foot of surface. These lengths are given in Table I. In the lighter wires, however, it will be found convenient to use some fraction of these lengths.

The method of making the test is very similar to that outlined for galvanized sheets, except that the wire is first cleaned with carbon tetrachloride or gasoline, and after being carefully weighed is placed in a tall glass cylinder containing hydrochloric acid (sp. gr. 1.20), to which has been added from 2 to 3 cc. of antimony-chloride solution of the same strength as used on galvanized sheets. The reason for using one-half the amount of antimony chloride in the case of wire is on account of taking one-half the area.

As previously stated, the coating on galvanized sheets in expressed in ounces per square foot, considering one side only, when in reality this amount of coating represents two square feet of surface. After immersing the entire length of wire for one minute it will be found convenient to pour the acid solution into another tall cylinder in order to facilitate removing the wire. The wire is then scrubbed under running water, wiped, thoroughly dried in a warm place for a few seconds and again weighed. Each gram lost corresponds to 1 oz. of coating per square foot. For direct comparison with the weight of coating as expressed on galvanized sheets, this figure should be doubled.

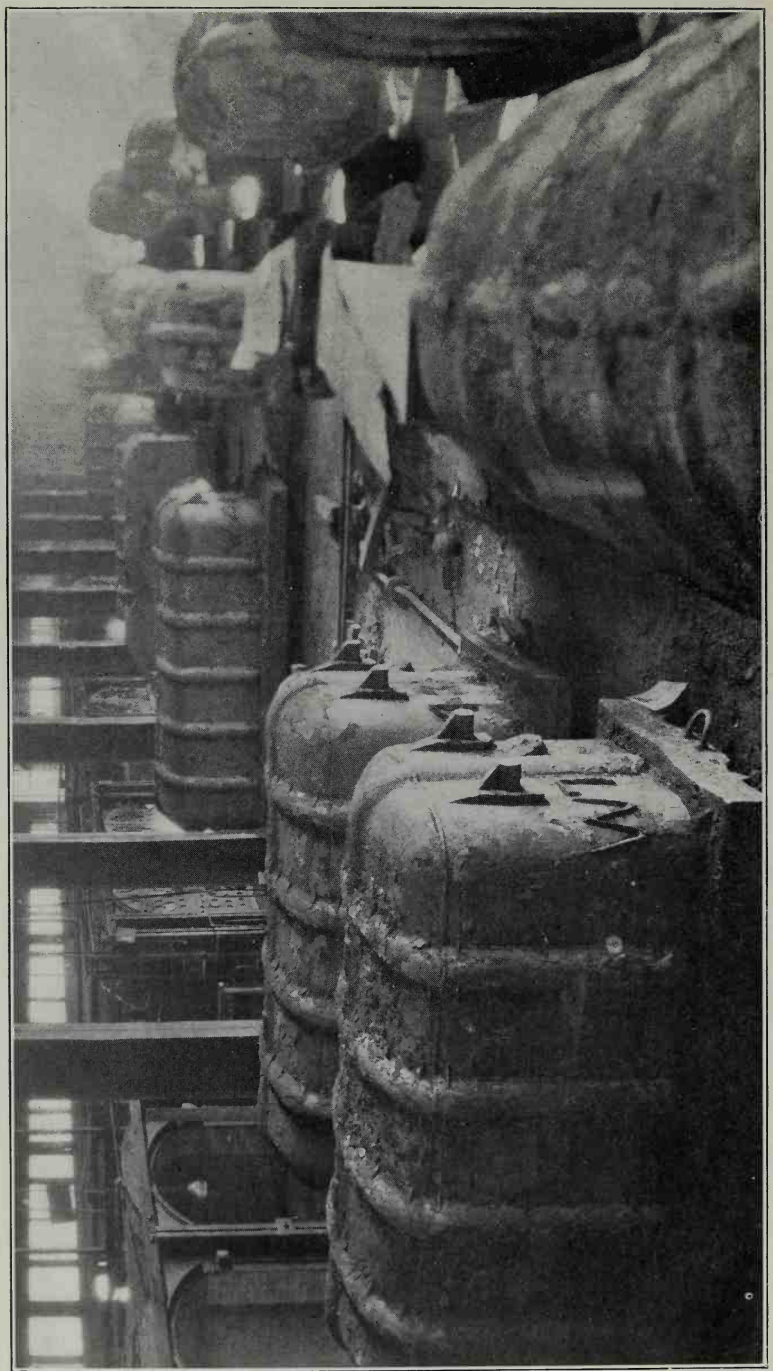


Note: For Commercial Testing, Squares A, B and C may be Used.
Numerals Represent Weight of Zinc Coating in ounces per square foot

TABLE I

LENGTHS OF WIRE TO GIVE GRAMS OF COATING EQUIVALENT TO OUNCES PER SQUARE FOOT

Gauge No.	Diameter, in.	Length for Test	
		in.	cm.
0	0.340	4–12/16	12.1
1	0.300	5– 6/16	13.7
2	0.284	5–11/16	14.5
3	0.259	6– 4/16	15.9
4	0.238	6–13/16	17.3
5	0.220	7– 6/16	18.7
6	0.203	7–15/16	20.2
7	0.180	9	22.8
8	0.165	9–13/16	24.9
9	0.148	10–15/16	27.7
10	0.134	12– 1/16	30.6
11	0.120	13– 8/16	34.2
12	0.109	14–13/16	37.7
13	0.095	17	43.2
14	0.083	19– 8/16	49.5
15	0.072	22– 7/16	57.0
16	0.065	24–14/16	63.2
17	0.058	27–14/16	70.8
18	0.049	33	83.8



General view in Annealing Department. At the left are shown the furnaces, while in the center are annealing covers ready for annealing and cooling after annealing. Two uncovered piles of sheets are also shown

DETERMINATION OF SPELTER COATING
LEAD ACETATE METHOD

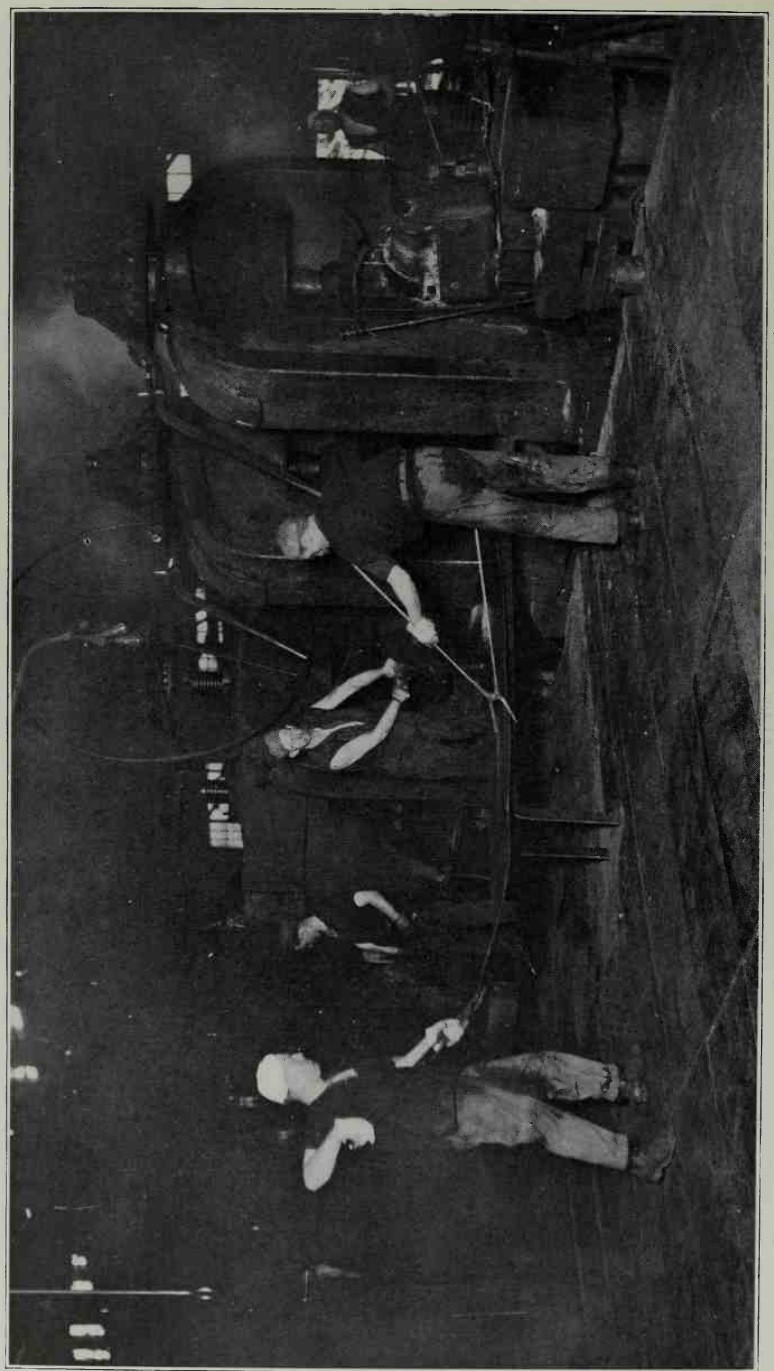
This test is based upon the fact that when a zinc coated article is placed in lead acetate at ordinary temperatures, the zinc passes into solution, and an equivalent amount of metallic lead is precipitated in a loosely adherent form upon the specimen. The reaction is retarded by the precipitation of the lead and, therefore, when a heavily galvanized piece is being tested, this lead must be periodically removed. The lead acetate solution can be used as the Preece copper sulphate test if desired. That is to measure the number of immersions which are required to remove all of the galvanized coatings. Should lead plate on the surface of the sample it is not easily confounded with the bright iron when exposed. The uncovering of the iron can be readily detected. The test is made as follows:

Three samples, 2¼ x 2¼″ are cut from a galvanized sheet as described in the previous method. The samples are weighed together and submerged separately for three minutes in tumblers containing the lead acetate solution. Tumblers are recommended on account of the fact that they are just the right diameter to enable the samples to be maintained in an upright position.

After submerging for three minutes the samples are taken out and the adherent lead removed with a stiff brush or steel spatula. A burnishing action should be avoided, as under some conditions closely adherent lead will be plated out on the iron. Repeat the three minute immersions in the lead acetate solutions until a bright surface is exposed. Four, 3-minute immersions are usually sufficient. Wash specimens in water, dry, warm slightly, allow to cool and weigh. The loss in grams divided by the number of samples taken represents the weight of coating in ounces per square foot.

The lead acetate solution is prepared by dissolving 400 grams of crystallized lead acetate in 1 liter of water. When dissolved, add 4 grams of finely powdered litharge and agitate until most of it has dissolved. The solution is allowed to settle and the clear portion decanted for use.

The Hydrochloric Acid Antimony Chloride Method is more reliable than this method, takes less time, and has been recommended as the standard method.



View showing a roughing and finishing stand in the sheet mill with a sheet ready to be put through the finishing stand. Sheets are rolled through the finishing stand two, four, six or eight at a time, depending upon the gauge and size desired. Sheet rolling is done entirely by hand and requires the highest degree of skill

SULPHUR

DETERMINATION OF SULPHUR BY EVOLUTION

Dissolve 5 grams of the sample in 100 cc. of hydrochloric acid, 1.10 specific gravity, contained in a 500 cc. flask fitted with rubber stopper containing thistle tube and educt tube, passing through a reflux condenser to prevent acid distilling over.

The educt tube dips almost to the bottom of a 10″x1″ test tube containing 50 cc. of cadmium chloride solution. A low flame is applied and flask heated until all metal has dissolved and all gas has been driven out of flask, as is evidenced by the steam condensing in cadmium chloride tube. The contents of test tube are washed into an 800 cc. beaker and sufficient water is added to bring the volume to 500 cc. Add 2 cc. of starch solution and 50 cc. of hydrochloric acid, 1.20 specific gravity. The solution is then titrated with standard iodine solution to blue color.

The standard iodine solution is prepared by dissolving 8.4 grams of iodine and 20 grams of potassium iodide in 50 cc. of distilled water. When iodine is in solution dilute to 2 liters and standardize with steel of known sulphur content. One cc. should equal .01% of sulphur when using 5 grams.

A solution of potassium iodate (KIO_3) can be used instead of the iodine and potassium iodide just described. The potassium iodate is more stable.

The Starch Solution can be prepared along any of the following lines:

(1) One gram arrowroot mixed in 10 cc. of cold water which is poured into 100 cc. of boiling water and immediately removed from the source of heat.

(2) Dissolve 20 grams of soluble starch in 100 cc. of distilled water to which can be added 40 grams of potassium iodide, free from iodine.

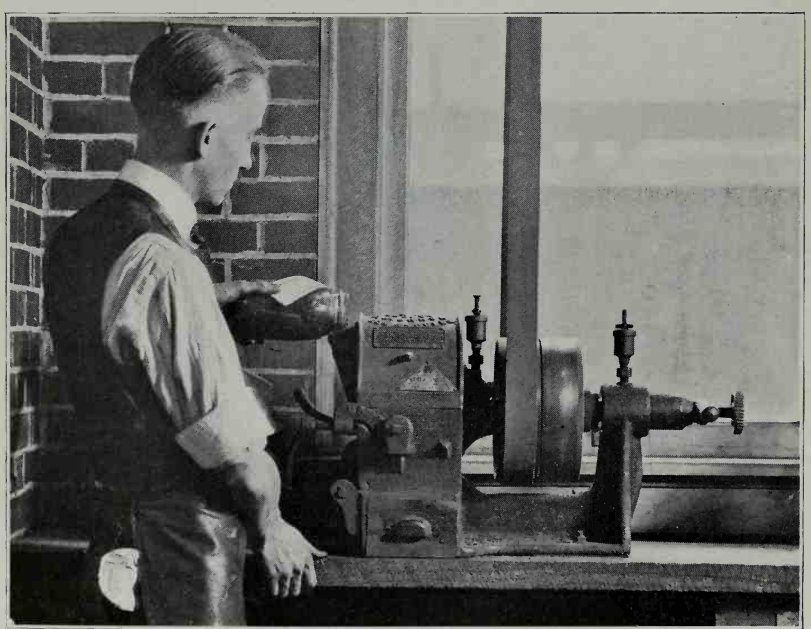
This mixture is then poured into 900 cc. of distilled water. Potassium iodide makes the starch more sensitive and it should not require

more than .2 of a cc. of iodine to give a permanent blue color in water containing a small amount of hydrochloric acid.

(3) If soluble starch is not available, corn starch can be used as follows:

To a mixture of 10 grams of corn starch and 50 cc. of distilled water, slowly add a solution containing 5 grams of caustic potash and 50 cc. of distilled water, until the starch changes to a clear paste. Dilute to 500 cc. with distilled water and add 10 grams of potassium iodide crystals, free from iodine.

(4) Prepare 500 cc. of a saturated solution of sodium chloride, and also a solution containing 100 cc. of 80% acetic acid in which 5 grams of starch has been dissolved. Pour into the sodium chloride solution and boil until clear. Make up to 600 cc. with distilled water, using 2 cc. for each determination of sulphur.



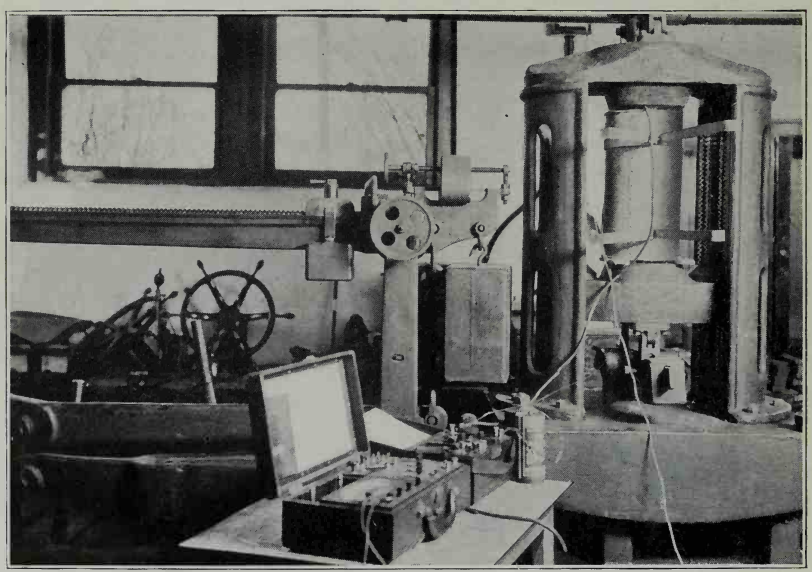
Chemist pulverizing sample of coal for chemical analysis

DETERMINATION OF SULPHUR
GRAVIMETRIC METHOD

We prefer the Gravimetric Method for the determination of sulphur where great accuracy is desired. We use the Bureau of Standards' method which is essentially as follows:

Dissolve the sample (4.57 grams) in 250 cc. of copper-potassium chloride solution (300 g. KCL-$CuCl_2$ and 100 cc. HCl per liter) and filter the residue on asbestos. Wash 2 or 3 times with 5% hydrochloric acid and then return residue and asbestos pad to the beaker and treat with 20 cc. of nitric acid (Sp. Gr. 1.42). Heat and add $KClO_3$ until all carbonaceous matter is destroyed.

Add a little (5 cc.) hydrochloric acid to dissolve the precipitated manganese dioxide and filter through asbestos again. Evaporate the solution to dryness, take up in 10 cc. of hydrochloric acid and evaporate to dryness again. Take up in 5 cc. of 2% hydrochloric acid and 20 cc. of water and filter through paper. Precipitate the sulphuric acid in the boiling filtrate with 2 cc. of hot 10% barium chloride solution. Digest a short time on the hot plate and filter. Wash the barium sulphate with water until free from chlorides, ignite slowly, and weigh. The weight of barium sulphate in grams multiplied by 3 is equal to the percentage of sulphur.



100,000 lb. Riehle Testing Machine, showing arrangements for carrying out tensile tests at high temperature

DETERMINATION OF SULPHUR
OXIDATION METHOD

In a 400 cc. beaker dissolve 5 grams of the steel in a mixture of 40 cc. of nitric acid, (Sp. Gr. 1.42) and 5 cc. of hydrochloric acid, (Sp. Gr. 1.20) add 0.5 grams of sodium carbonate and evaporate the solution to dryness. Add 40 cc. of hydrochloric acid, (Sp. Gr. 1.20) evaporate to dryness and bake at a moderate heat. After solution of the residue in 30 cc. of hydrochloric acid, (Sp. Gr. 1.20) and evaporation to syrupy consistency, add 2 to 4 cc. of hydrochloric acid (Sp. Gr. 1.20), and then 30 to 40 cc. of hot water.

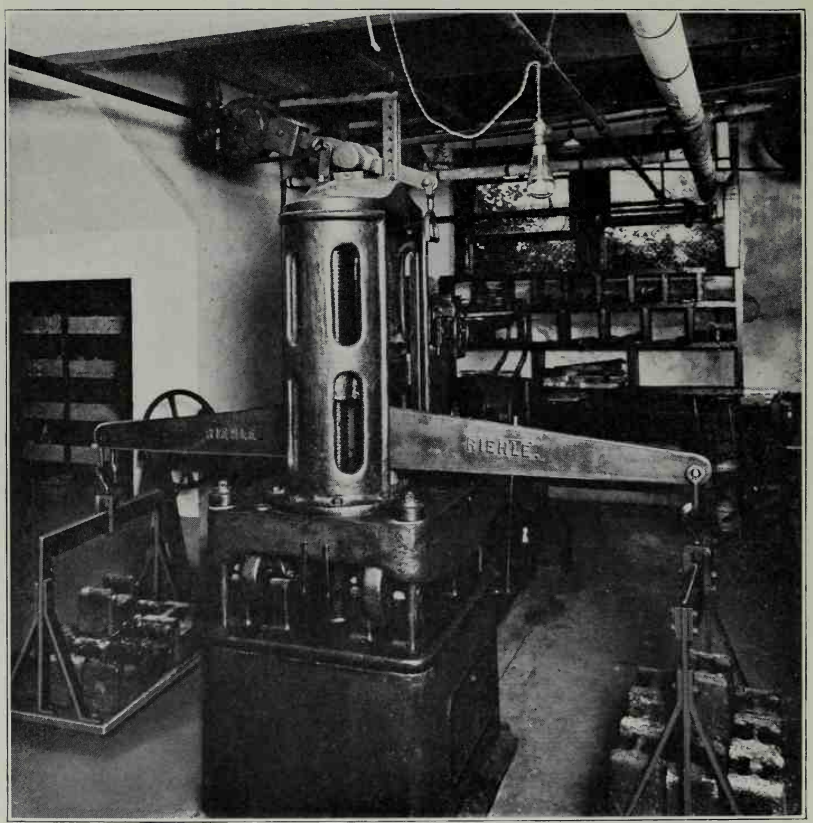
Filter and wash with cold water, the final volume not exceeding 100 cc. To the cold filtrate add 10 cc. of the barium chloride solution. Let stand at least 24 hours, filter on a 9-cm. paper, wash the precipitate first with a hot solution containing 10 cc. of hydrochloric acid, (Sp. Gr. 1.20), and 1 gram barium chloride to the liter, until free from iron; and then with hot water till free from chloride. Ignite and weigh as barium sulphate.

Keep the washings separate from the main filtrate and evaporate them to recover any dissolved barium sulphate.

NOTE: A blank determination on all reagents used should be made and the results corrected accordingly.

Barium Chloride

Dissolve 100 grams of barium chloride in 1000 of distilled water.



Physical Testing Machine capacity 100,000 lbs., illustrating method used for checking the accuracy of the machine

TIN PLATE

METHOD FOR SAMPLING AND ANALYSIS OF TIN, TERNE AND LEAD-COATED SHEETS

Four 2 by 4-in. pieces are cut, one from each end and each side of the sheet, parallel with the sides and equidistant from the ends, as shown on Page 198. One sheet from each grade or shipment is taken for analysis.

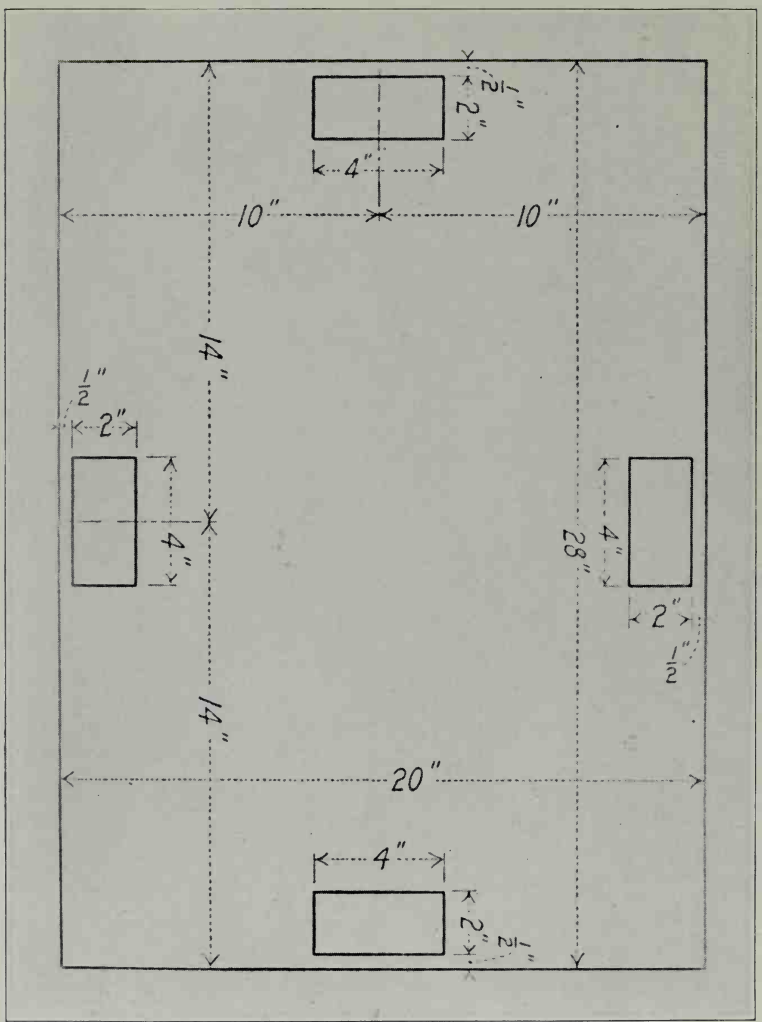
These samples, before weighing, should be thoroughly cleaned with chloroform, carbon tetrachloride or gasoline. Each piece is then cut in half, marking one half "A" and the other half "B". The four pieces comprising lot A are then accurately weighed together, cut into small pieces about $\frac{1}{8}$ in. square, thoroughly mixed, and used for the determination of tin and lead. The four pieces comprising lot B are reserved for the analysis of base metal and the direct determination of coating as a check on the analysis of lot A.

A templet should be provided, made preferably from steel $\frac{1}{8}$ in. thick and exactly 2 by 4 in. A scribe is used to accurately mark the sections to be cut. The templet is then used to subdivide the 2 by 4 in. specimens into two pieces, 2 by 2 in. The sections for analysis are then cut with tinner's shears.

METHOD OF ANALYSIS

DETERMINATION OF TIN

Three 5-gram portions of the finely cut sample of lot A are placed into three 300 cc. Erlenmeyer flasks, each fitted with a one-hole rubber stopper containing a glass tube bent twice at right angles, one end of which projects through the rubber stopper for a short distance, the other end being long enough to reach almost to the bottom of a beaker, placed on a level with the flask, containing about 300 cc. of dilute sodium-bicarbonate solution. Add 75 cc. of concentrated hydrochloric acid, connect the flask with the stopper containing the glass tube, and place the flask on a hot-plate. Heat gradually at first until most of the metal is in solution. The long end of the glass tube, in the meantime, is submerged in the beaker.


2½"
2"
4"
10"
10"
14"
½"
2"
4"
28"
4"
2"
½"
14"
20"
4"
2"
2½"

The hydrochloric acid solution is finally brought to boiling and when all the metal is dissolved the beaker containing dilute sodium-bicarbonate solution is replaced by one containing a saturated solution of the same. Remove the beaker and flask to a cool place. This will cause a small amunt of the sodium-bicarbonate to enter the flask and exclude the air. The solution is finally brought to a low temperature, preferably with ice water. This solution is then diluted to about 200 cc. with oxygen-free water which contains several cubic centimeters of starch solution, and titrated with $N/20$ iodine solution. We have found this strength of iodine solution to be the most satisfactory for this method.

The distilled water free from oxygen is obtained in any of three ways: (1) By passing carbon dioxide through the cold distilled water; (2) By boiling vigorously and cooling; or (3) by adding a few cubic centimeters of concentrated hydrochloric acid to the water and then about 2 g. of sodium bicarbonate stirring vigorously. By running this determination in triplicate, the first titration serves as a control to indicate the number of cubic centimeters of iodine required, whence the two succeeding titrations may be made very rapidly and should check very closely.

Standardizing the Iodine Solution:

About 0.1 g. of pure tin and 4 g. of iron filings are dissolved in 75 cc. of concentrated hydrochloric acid, etc., as under the determination of tin. One cubic centimeter of $N/20$ iodine = 0.002975 g. of tin.

Calculation—Weight of tin:

$$\frac{\text{Wt. of tin on 5 g.} \times \text{Wt. (g.) of 16 sq. in.}}{5} \times 8.6421 = \text{number of}$$

pounds per case of 112 sheets, 20 by 28 in.

DETERMINATION OF LEAD

Dissolve 10 g. of the finely cut sample of lot A in 150 cc. of nitric acid (1:1). Heat until free from brown fumes and dilute to 1 liter and mix thoroughly. Take 100 cc. of this solution, add 10 cc. of concentrated nitric acid, electrolyze at a temperature of 50 to 60° C., using 1 to 2 amperes and 2.3 to 2.5 volts. The weight of PbO_2 is multiplied by 0.866.

If the base metal contains an appreciable amount of manganese the lead should be determined as sulfate.

Calculation—Weight of lead:

$$PbO_2 \text{ found (g.)} \times 0.866 \times 10 = Pb;$$

$$\frac{Pb \times \text{Wt. (g.) of 16 sq. in.}}{10} \times 8.6421 = \text{number of pounds}$$

per case of 112 sheets, 20 by 28 in.

DIRECT DETERMINATION OF THE WEIGHT OF COATING

The remaining four pieces representing lot B are used for the analysis of the base metal and incidentally can be used for the direct determination of the weight of coating.

The four 2 by 2-in. pieces are carefully weighed together and each piece is wrapped with a stiff platinum or nickel wire in such a manner that it may be placed in the acid in a horizontal position. Heat 60 cc. of concentrated sulphuric acid contained in a 400-cc Jena glass beaker to at least 250° C., immerse each piece separately in the hot acid for exactly 1 minute, and remove to a 600-cc. Jena beaker containing 50 cc. of distilled water. Immerse momentarily and rub the surface while washing with about 50 cc. more of distilled water, using a wash bottle for this purpose. The four samples are thoroughly dried, reweighed, and used for the analysis of base metal.

The loss in weight represents the coating and some iron. The sulphuric acid contained in the 400 cc. beaker is cooled and combined with the washings in the 600 cc. beaker. Two hundred cubic centimeters of concentrated hydrochloric acid are added and the solution boiled for a few minutes. The solution is cooled, poured into a graduated 500 cc. flask and filled to the mark with distilled water.

DETERMINATION OF IRON

Place 100cc. of this solution in a 300cc. Erlenmeyer flask, add 1 cc. of a standard solution of potassium permanganate to oxidize the iron and tin, heat to boiling and reduce with a few drops of stannous chloride. Cool, pour into a liter beaker containing 400 cc. of distilled water, add 25 cc. of mercuric chloride, followed by 10 cc. of phosphoric acid and manganese-sulphate solution, and titrate with $N/10$ potassium permanganate.

Calculation—

Four pieces 2 by 2 in. weigh	28.5686 g.
Same after stripped in acid	24.1620 g.
Loss, coating plus iron	4.4066 g.
Iron as found by titration	0.4887 g.
Weight of coating	3.9179 g.

3.9179 x 8.6421 = number of pounds per case of 112 sheets, 20 by 28 in.

$$\frac{\text{Tin in 100 cc. x 5 x 100}}{\text{Weight of Coating}} = \text{percentage of tin.}$$

Percentage of lead is obtained by difference.

In the analysis of tin plate, the weight of coating is expressed in pounds per box, which is a half case, or 112 sheets 14 by 20 in.; hence to obtain the weight of coating per box on tin plate, the number of pounds as obtained above is divided by two.

CHECK DETERMINATION OF TIN

The remainder of the solution which has been used for the determination of iron can be used for the determination of tin as follows: Place three portions of 100 cc. each in three 300 cc. Erlenmeyer flasks. If any of the lead sulphate should or should not be removed in any of these portions, the accuracy of the tin determination is not affected. Add 1 g. of powdered antimony, connect with rubber stopper and glass tube described in the method of determination of tin in the sample of lot A, place on a hot-plate, using dilute sodium biarbonate solution as a trap, and boil until the solution becomes decolorized. Replace the dilute sodium-bicarbonate solution with a saturated solution of the same, remove from the hot-plate, cool, dilute and complete the determination as described under the first method.

CONCLUSIONS

We claim for this method that the sample shows a true average of the coating on the plate, since we have checked the coating very closely by this method and by sampling from the center of the sheet, even with such large samples as 10 by 10 in. When 5 g. of the sample are taken for the determination of tin, an area of about 2.5 sq. in. is represented in the case of 40-lb., IC plate, and of about 3 sq. in. in the case of 25-lb. plate; while, of course, it is double this in the determination of lead. Furthermore, the amount of sample taken here for

analysis is a representative quantity from 16 sq. in. and not merely from one particular section of 2.5 of 3 sq. in.; and is as large as many laboratories are using and larger than most are using.

In addition, we believe that this method is more truly average than any method we have investigated, and moreover, the sheet is not destroyed so far as usefulness is concerned, but may be sheared down to a smaller size. While it is not necessary to determine the weight of coating directly by the sulphuric acid method, in addition to the determination of the lead and tin (on lot A), it will, however, serve as a check, and should agree very closely with it. Furthermore, this is an excellent method for stripping the coating preliminary to the analysis of the base metal.

By running the determination of tin in triplicate, as described, the method is very rapid and accurate, whereas the method as now used by many laboratories in which the plate is dissolved in an atmosphere of carbon dioxide in a graduated flask, cooled, diluted to volume and titrated in aliquots, involves many details and is not so rapid. In this method also, no antimony is needed for the reduction of tin, since the iron in the base metal accomplishes this; moreover, in the presence of the quantity of tin here involved the antimony would have a tendency to deposit back on the plate, retarding the solution of the tin and thus giving low results.

With the use of a rotating anode the proposed method is very rapid and the entire determination can be finished in a reasonable length of time.

TITANIUM

BUREAU OF STANDARDS' METHOD FOR THE DETERMINATION OF TITANIUM

Titanium is determined by treating 5 grams of iron with 40 cc. of hydrochloric acid (1:1) and heating until all iron has gone into solution. Dissolving in this manner, all but a negligible quantity of titanium remains in the insoluble residue. The filtrate is tested for titanium by extracting the iron with ether after oxidation with a small amount of nitric acid, using the method of Rothe (Stahl und Eisen, 12, 1052 (1892), and 13, 333 (1893),) and adding hydrogen peroxide to the extracted solution, after expelling the ether and oxidizing with nitric acid. In all cases only a faint coloration is obtained. The insoluble residue is filtered off and washed with hot water, and the filter paper and carbonaceous matter are burned.

The residue in the crucible is treated with hydrofluoric acid and a little sulphuric acid, and all silicon volatilized. The residue is fused with sodium carbonate, treated with water, and acidified with sulphuric acid.

A sufficient amount of ferric alum is added to the standard titanium solution to give the same tint as the sample when they are at the same time dilution, for it is found that the residue from the silica always contains a little iron along with the titanium. Hydrogen peroxide is added to the solution and standard and the comparison made in a Wolff colorimeter.

Reagents

Peroxide Solution:

Dissolve 4 grams of sodium peroxide in 125 cc. dilute sulphuric acid (1 of acid to 3 of water), and dilute to 500 cc.

Concentrated Standard Titanium Solution:

One-fourth gram of a standard 20% carbonless ferro-titanium is dissolved in 30 cc. of dilute sulphuric acid (1 acid to 3 water). When solution is complete it is oxidized by the least possible quantity of concentrated nitric acid, boiled for a few minutes, cooled and diluted to

such a volume that 1 cc. will contain 0.0005 gram of titanium. When using a five-gram sample 1 cc. is therefore equal to 0.01% titanium.

Dilute Standard Titanium Solution:

This solution is made, just before making the determination, by diluting one volume of the concentrated standard titanium solution to ten volumes.

One cc. of this solution contains 0.00005 gram of titanium and is equal to 0.001% of titanium when using a 5-gram sample.

VANADIUM

DOUGHERTY'S[1] METHOD FOR THE DETERMINATION OF VANADIUM IN STEEL

In the application of Johnson's[2] or similar methods for the determination of vanadium in steel, considerable difficulty is often experienced in producing a colorless or "old rose" shade with ferrous sulphate in the solution containing an excess of permanganate after the preliminary oxidation of the vanadium. To obviate this difficulty the following method has been developed, in which this oxidation of the vanadium is effected by a sufficient quantity of nitric acid alone or with ammonium persulphate.

Method

Treat 2 to 4 grams of the drillings in a 500 cc. Erlenmeyer flask, with 60 cc. of water and 10 cc. of concentrated sulphuric acid. After heating the solution nearly to boiling, until the reaction is complete, add 40 cc. of nitric acid (Sp. Gr. 1.20), and boil thoroughly for 10 minutes to oxidize the iron and vanadium and to expel the last traces of nitrous fumes.

Cool the solution, add 60 cc. of cold sulphuric acid (1:2) and dilute in a 600 cc. beaker to 450 cc. Add 3 cc. of a freshly prepared 1% solution of potassium ferricyanide, and titrate rather rapidly, with constant stirring, with $N/20$ ferrous ammonium sulphate, to the appearance of the first dark blue color. The end point can best be observed by looking through the side of the beaker toward the bottom of the beaker placed directly before a window. Deduction of a blank of 0.4 cc. of the ferrous solution has been found necessary, and is independent of the weight of the sample, the presence of chromium, and of the carbon content up to 0.5 per cent.

For steels with over 0.50 per cent carbon, the blanks are higher; and, moreover, with 4-gram samples of such steels, the end point is rendered indistinct by a turbidity which appears toward the end of the titration. This difficulty may be avoided by adding to the solution immediately after the boiling with nitric acid as above, 60 cc. of 1:2 sulphuric acid and 5 to 8 grams of ammonium persulphate (which in the absence of silver nitrate will not oxidide the Cr. and Mn.), and continuing to boil for 15 minutes, so that all nitrous oxides and hydrogen peroxide are expelled. (Before this second boiling,

1 George T. Dougherty, The Journal of Industrial and Engineering Chemistry, May, 1915.
2 C. M. Johnson, "Analysis of Special Steels."

wash down with hot water the persulphate which sticks to the glass.) Cool, dilute and titrate as above. After such treatment the blank is .35 cc. (instead of .4 cc.) for steels with under .5 per cent carbon, and .5 cc. for .60 to .70 carbon, and .6 cc. for .90 to 1.25 carbon steels.

The blanks are the same with or without the persulphate treatment for steels of over .50 per cent carbon.

The ferrous ammonium sulphate solution may be standardized against $N/10$ permanganate, the strength of which has been determined with sodium oxalate. The iron value of the permanganate multiplied by .917 gives the vanadium value.

If chromium is desired it should be determined on a separate portion, using the sodium bismuthate oxidation method.

USEFUL DATA

To find circumference of a circle multiply diameter by 3.1416.
To find diameter of a circle multiply circumference by .31831.
To find area of a circle multiply square of diameter by .7854.
To find area of a triangle multiply base by ½ perpendicular height.
To find surface of a sphere multiply square of diameter by 3.1416.
To find solidity of a sphere multiply cube of diameter by .5236.
Doubling the diameter of a pipe increases its capacity four times.

A gallon of water (U. S. Standard) weighs 8 lbs. ⅓ oz., and contains 231 cubic inches.

A cubic foot of water contains 1728 cubic inches, 7½ gallons and weighs 62½ pounds.

A standard horse power: The evaporation of 30 pounds of water per hour from a feed water temperature of 100 deg. F., into steam at 70 pounds gauge pressure.

To find capacity of tanks any size; given dimensions of a cylinder in inches, to find its capacity in U. S. gallons: Square the diameter, multiply by the length and by .0034.

1 meter = 39.37 inches.

2.54 cm. = 1 inch

28316 cc. = 1 cubic foot.

29.573 cc = 1 fluid oz.

1000 cc. = 1.05668 quarts.

3785.43 cc. = 1 U. S. Gallon (231 cu. in.)

1 gram = 15.4324 grains = .035274 oz. avoirdupois.

1 kilo = 2.2046 pounds (avoirdupois).

28.35 grams = 1 oz.

453.59 grams = 1 pound.

CONVERSION TABLES OF FAHRENHEIT AND CENTIGRADE SCALES

Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.
0	32	200	392	400	752	600	1112	800	1472
5	41	205	401	405	761	605	1121	805	1481
10	50	210	410	410	770	610	1130	810	1490
15	59	215	419	415	779	615	1139	815	1499
20	68	220	428	420	788	620	1148	820	1508
25	77	225	437	425	797	625	1157	825	1517
30	86	230	446	430	806	630	1166	830	1526
35	95	235	455	435	815	635	1175	835	1535
40	104	240	464	440	824	640	1184	840	1544
45	113	245	473	445	833	645	1193	845	1553
50	122	250	482	450	842	650	1202	850	1562
55	131	255	491	455	851	655	1211	855	1571
60	140	260	500	460	860	660	1220	860	1580
65	149	265	509	465	869	665	1229	865	1589
70	158	270	518	470	878	670	1238	870	1598
75	167	275	527	475	887	675	1247	875	1607
80	176	280	536	480	896	680	1256	880	1616
85	185	285	545	485	905	685	1265	885	1625
90	194	290	554	490	914	690	1274	890	1634
95	203	295	563	495	923	695	1283	895	1643
100	212	300	572	500	932	700	1292	900	1652
105	221	305	581	505	941	705	1301	905	1661
110	230	310	590	510	950	710	1310	910	1670
115	239	315	599	515	959	715	1319	915	1679
120	248	320	608	520	968	720	1328	920	1688
125	257	325	617	525	977	725	1337	925	1697
130	266	330	626	530	986	730	1346	930	1706
135	275	335	635	535	995	735	1355	935	1715
140	284	340	644	540	1004	740	1364	940	1724
145	293	345	653	545	1013	745	1373	945	1733
150	302	350	662	550	1022	750	1382	950	1742
155	311	355	671	555	1031	755	1391	955	1751
160	320	360	680	560	1040	760	1400	960	1760
165	329	365	689	565	1049	765	1409	965	1769
170	338	370	698	570	1058	770	1418	970	1778
175	347	375	707	575	1067	775	1427	975	1787
180	356	380	716	580	1076	780	1436	980	1796
185	365	385	725	585	1085	785	1445	985	1805
190	374	390	734	590	1094	790	1454	990	1814
195	383	395	743	595	1103	795	1463	995	1823

CONVERSION TABLES OF FAHRENHEIT AND CENTIGRADE SCALES

Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.	Cent.	Fahr.
1000	1832	1190	2174	1380	2516	1570	2858
1005	1841	1195	2183	1385	2525	1575	2867
1010	1850	1200	2192	1390	2534	1580	2876
1015	1859	1205	2201	1395	2543	1585	2885
1020	1868	1210	2210	1400	2552	1590	2894
1025	1877	1215	2219	1405	2561	1595	2903
1030	1886	1220	2228	1410	2570	1600	2912
1035	1895	1225	2237	1415	2579	....	....
1040	1904	1230	2246	1420	2588	....	....
1045	1913	1235	2255	1425	2597	....	....
1050	1922	1240	2264	1430	2606	....	....
1055	1931	1245	2273	1435	2615	....	....
1060	1940	1250	2282	1440	2624	....	....
1065	1949	1255	2291	1445	2633	....	....
1070	1958	1260	2300	1450	2642	....	....
1075	1967	1265	2309	1455	2651	....	....
1080	1976	1270	2318	1460	2660	....	....
1085	1985	1275	2327	1465	2669	....	....
1090	1994	1280	2336	1470	2678	....	....
1095	2003	1285	2345	1475	2687	....	....
1100	2012	1290	2354	1480	2696	....	....
1105	2021	1295	2363	1485	2705	....	....
1110	2030	1300	2372	1490	2714	....	....
1115	2039	1305	2381	1495	2723	....	....
1120	2048	1310	2390	1500	2732	....	....
1125	2057	1315	2399	1505	2741	....	....
1130	2066	1320	2408	1510	2750	....	....
1135	2075	1325	2417	1515	2759	....	....
1140	2084	1330	2426	1520	2768	....	....
1145	2093	1335	2435	1525	2777	....	....
1150	2102	1340	2444	1530	2786	....	....
1155	2111	1345	2453	1535	2795	....	....
1160	2120	1350	2462	1540	2804	....	....
1165	2129	1355	2471	1545	2813	....	....
1170	2138	1360	2480	1550	2822	....	....
1175	2147	1365	2489	1555	2831	....	....
1180	2156	1370	2498	1560	2840	....	....
1185	2165	1375	2507	1565	2849	....	....

CENTIGRADE AND FAHRENHEIT CONVERSIONS

To change Centigrade Temperatures to Fahrenheit Temperatures—Add 40° multiply by 1.8, then subtract 40°.

To change Fahrenheit Temperatures to Centigrade Temperatures—Add 40° multiply by .5555 (5/9), then subtract 40°.

MELTING POINTS OF THE CHEMICAL ELEMENTS

Element	C	F	Element	C	F	Element	C	F
Helium	> —271	> —456	CADMIUM	320.9	609.6	Cobalt	1480	2696
Hydrogen	—259	—434	LEAD	327.4	621.3	Yttrium	1490	2714
Neom	—253?	—423	ZINC	419.4	786.9	IRON	1530	2786
Fluorine	—223	—369	Tellurium	452	846	PALLADIUM	1550	2822
Oxygen	—218	—360	ANTIMONY	630.0	1166.0	Chromium	1615	2939
Nitrogen	—210	—346	Cerium	640	1184	Zicronium	1700?	3090
Argon	—188	—306	Magnesium	651	1204	Columbium		
Krypton	—169	—272	ALUMINUM	658.7	1217.7	(Niobium)	1700?	3090
Xenon	—140	—220	Radium	700	1292	Thorium	> 1700	> 3009
Chlorine	—101.5	—150.7	Calcium	810	1490		< Mo.	< Mo.
MERCURY	— 38.87	— 37.97	Lanthanum	810?	1490	Vanadium	1720	3128
Bromine	— 7.3	+ 18.9	Strontium	> Ca < Ba ?	.....	PLATINUM	1755	3191
Caesium	+ 26	79	Neodymium	840?	1544	Ytterbium	?	.....
Gallium	30	86	Arsenic	850	1562	Titanium	1800	3272
Rubidium	38	100	Barium	850	1562	Uranium	< 1850	< 3360
Phosphorus	44	111	Praseodymium	940	1724	Rhodium	1950	3542
Potassium	62.3	144.1	Germanium	958	1756	Boron	2200–2500?	4000–4500
Sodium	97.5	207.5	SILVER	960.5	1760.9	Iridium	2350?	4260
Iodine	113.5	236.3	GOLD	1063.0	1945.5	Ruthenium	2450?	4440
	S_I 112.8	235.0	COPPER	1083.0	1981.4	Molybdenum	2550	4620
Sulphur	S_{II} 119.2	246.6	Manganese	1230	2246	Osmium	2700?	4890
	S_{III} 106.8	224.2	Beryllium			Tantalum	2900	5250
Indium	155	311	(Glucinum)	1280	2336	TUNGSTEN	3400	6152
Lithium	186	367			> 2370–	Carbon	> 3600	> 6500
Selenium	217–220	423–428	Samarium	1300–1400	< 2550			
TIN	231.9	449.4	Scandium	?	.....			
Bismuth	271	520	Silicon	1420	2588			
Thallium	302	576	NICKEL	1452	2646			

OTHER STANDARD TEMPERATURES

Substance	Phenomenon	C	F	Variation with pressure (pressure in mm. of Hg.)
OXYGEN	Boiling	—183.0	—297.4	$C° = -183.0 + 0.01258(p - 760) - 0.0000079(p - 760)^2$
CARBON DIOXIDE	Sublimation	— 78.5	—109.3	$C° = -78.5 + 0.01595(p - 760) - 0.0000111(p - 760)^2$
SODIUM SULPHATE $Na_2SO_4 + 10H_2O$	Transformation into anhydrous salt	32.384	90.29	
WATER	Boiling	100	212	$C° = 100 + 0.03670(p - 760) - 0.00002046(p - 760)^2$
NAPHTHALENE	do	217.96	424.33	$C° = 217.96 + 0.058(p - 760)$
BENZOPHENONE	do	305.9	582.6	$C° = 305.9 + 0.063(p - 760)$
SULPHUR	do	444.6	832.3	$C° = 444.6 + 0.0908(p - 760) - 0.000047(p - 760)^2$
71.9 per cent Ag 28.1 per cent Cu	Eutectic freezing	779	1434	
SODIUM CHLORIDE	Freezing	801	1474	

PERIODIC CLASSIFICATION OF THE ELEMENTS

ON BASIS O = 16

REVISED AND CORRECTED TO 1918

Series	Group 0	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7	Group 8
1		H=1.008							
2	He=4.00	Li=6.94	Gl=9.1	B=11.0	C=12.005	N=14.01	O=16.00	F=19.0	
3	Ne=20.2	Na=23.00	Mg=24.32	Al=27.1	Si=28.3	P=31.04	S=32.06	Cl=35.46	
4	A=39.88	K=39.10	Ca=40.07	Sc=44.1	Ti=48.1	V=51.0	Cr=52.0	Mn=54.93	Fe=55.84 Co=58.97 Ni=58.68
5		Cu=63.57	Zn=65.37	Ga=69.9	Ge=72.5	As=74.96	Se=79.2	Br=79.92	
6	Kr=82.92	Rb=85.45	Sr=87.63	Yt=88.7	Zr=90.6	Cb=93.1	Mo=96.0		Ru=101.7 Rh=102.9 Pd=106.7
7		Ag=107.88	Cd=112.4	In=114.8	Sn=118.7	Sb=120.2	Te=127.5	I=126.92	
8	Xe=130.2	Cs=132.81	Ba=137.37	La=139.0	Ce=140.25	Pr=140.9	Nd=144.3		Sa=150.4 Eu=152.0 Gd=157.3
9		——	——	Er=167.7	——	Yb=173.5	——	——	
10	——	——	——	——	——	Ta=181.5	W=184.0	——	Os=190.9 Ir=193.1 Pt=195.2
11		Au=197.2	Hg=200.6	Tl=204.0	Pb=207.20	Bi=208.0			
12	Nt=222.4		Ra=226.0		Th=232.4		U=238.2		

RARE EARTH METALS NOT PLACED:– Dy-162.5. Ho-163.5. Lu-175.0. Tb-159.2. Tm-168.5.

Permission of Central Scientific Co., Chicago

AN ACT ESTABLISHING A STANDARD GAGE FOR SHEET AND PLATE IRON AND STEEL

Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled, That for the purpose of securing uniformity, the following is established as the only standard gage for sheet and plate iron and steel in the United States of America, namely:

Number of gage	Approximate thickness in fractions of an inch	Approximate thickness in decimal parts of an inch	Approximate thickness in millimeters	Weight per square foot in ounces avoirdupois	Weight per square foot in pounds avoirdupois	Weight per square foot in kilograms	Weight per square meter in kilograms	Weight per square meter in pounds avoirdupois
0000000	1–2	.5	12.7	320	20.0	9.0	97.	215.
000000	15–32	.46	11.9	300	18.7	8.5	91.	201.
00000	7–16	.43	11.1	280	17.5	7.9	85.	188.
0000	13–32	.40	10.3	260	16.2	7.3	79.	174.
000	3–8	.375	9.5	240	15	6.8	73.	161.
00	11–32	.343	8.7	220	13.7	6.2	67.	148.
0	5–16	.312	7.9	200	12.5	5.6	61.	134.
1	9–32	.281	7.1	180	11.2	5.1	54.	121.
2	17–64	.265	6.7	170	10.6	4.8	51.	114.
3	1–4	.25	6.3	160	10	4.5	48.	107.
4	15–64	.234	5.9	150	9.3	4.2	45.	100.
5	7–32	.218	5.5	140	8.7	3.96	42.	94.
6	13–64	.203	5.1	130	8.1	3.68	39.6	87.
7	3–16	.187	4.7	120	7.5	3.40	36.6	80.
8	11–64	.171	4.3	110	6.8	3.11	33.5	74.
9	5–32	.156	3.96	100	6.2	2.83	30.5	67.
10	9–64	.140	3.57	90	5.6	2.55	27.4	60.
11	1–8	.125	3.17	80	5	2.26	24.4	53.
12	7–64	.109	2.77	70	4.3	1.98	21.3	47.
13	3–32	.093	2.38	60	3.75	1.70	18.3	40.
14	5–64	.078	1.98	50	3.12	1.41	15.2	33.6
15	9–128	.070	1.78	45	2.81	1.27	13.7	30.2
16	1–16	.062	1.58	40	2.5	1.13	12.2	26.9
17	9–160	.056	1.42	36	2.25	1.02	10.9	24.2
18	1–20	.05	1.27	32	2	.90	9.7	21.5
19	7–160	.043	1.11	28	1.75	.79	8.5	18.8
20	3–80	.0375	.95	24	1.50	.68	7.3	16.1
21	11–320	.0343	.87	22	1.37	.62	6.7	14.8
22	1–32	.0312	.79	20	1.25	.56	6.1	13.4
23	9–320	.0281	.71	18	1.12	.51	5.4	12.1
24	1–40	.025	.63	16	1	.45	4.8	10.7
25	7–320	.0218	.55	14	.87	.396	4.2	9.4
26	3–160	.0187	.47	12	.75	.340	3.66	8.0
27	11–640	.0171	.43	11	.68	.311	3.35	7.4
28	1–64	.0156	.396	10	.62	.283	3.05	6.7
29	9–640	.0140	.357	9	.56	.255	2.74	6.0
30	1–80	.0125	.317	8	.5	.223	2.44	5.3
31	7–640	.0109	.277	7	.43	.198	2.13	4.7
32	13–1280	.0101	.257	6½	.40	.184	1.98	4.3
33	3–320	.0093	.238	6	.375	.170	1.83	4.0
34	11–1280	.0085	.218	5½	.343	.155	1.67	3.70
35	5–640	.0078	.198	5	.312	.141	1.52	3.36
36	9–1280	.0070	.178	4½	.281	.127	1.37	3.03
37	17–2560	.0066	.168	4¼	.265	.120	1.29	2.87
38	1–160	.0062	.158	4	.25	.113	1.22	2.69

And on and after July first, eighteen hundred and ninety-three, the same and no other shall be used in determining duties and taxes levied by the United States of America on sheet and plate iron and steel. But this act shall not be construed to increase duties upon any articles which may be imported.

Sec. 2. That the Secretary of the Treasury is authorized and required to prepare suitable standards in accordance herewith.

Sec. 3. That in the practical use and application of the standard gage hereby established a variation of two and one-half per cent either way may be allowed.

Approved, March 3, 1893.

ELECTROCHEMICAL SERIES

POSITION IN ELECTROCHEMICAL SERIES OF VARIOUS SUBSTANCES, IN THE ORDER OF THE MOST POSITIVE FIRST

1 Caesium	17 Nickel	33 Rhodium
2 Rubidium	18 Thallium	34 Platinum
3 Potassium	19 Indium	35 Osmium
4 Sodium	20 Lead	36 Silicon
5 Lithium	21 Cadmium	37 Carbon
6 Barium	22 Tin	38 Boron
7 Strontium	23 Bismuth	39 Nitrogen
8 Calcium	24 Copper	40 Arsenic
9 Magnesium	25 Hydrogen	41 Selenium
10 Aluminum	26 Mercury	42 Phosphorus
11 Chromium	27 Silver	43 Sulphur
12 Manganese	28 Antimony	44 Iodine
13 Zinc	29 Tellurium	45 Bromine
14 Gallium	30 Palladium	46 Chlorine
15 Iron	31 Gold	47 Oxygen
16 Cobalt	32 Iridium	48 Fluorine

All elements preceding iron are electro-positive to iron. All following iron are electro-negative.

ATOMIC WEIGHTS

INTERNATIONAL ATOMIC WEIGHTS, 1918

	Symbol	Atomic Weight
Aluminium	Al	27.1
Antimony	Sb	120.2
Argon	A	39.88
Arsenic	As	74.96
Barium	Ba	137.37
Bismuth	Bi	208.0
Boron	B	11.0
Bromine	Br	79.92
Cadmium	Cd	112.40
Caesium	Cs	132.81
Calcium	Ca	40.07
Carbon	C	12.05
Cerium	Ce	140.25
Chlorine	Cl	35.46
Chromium	Cr	52.0
Cobalt	Co	58.97
Columbium	Cb	93.1
Copper	Cu	63.57
Dysprosium	Dy	162.5
Erbium	Er	167.7
Europium	Eu	152.0
Fluorine	F	19.0
Gadolinium	Gd	157.3
Gallium	Ga	69.9
Germanium	Ge	72.5
Glucinum	Gl	9.1
Gold	Au	197.2
Helium	He	4.0
Holmium	Ho	163.5
Hydrogen	H	1.008
Indium	In	114.8
Iodine	I	126.92
Iridium	Ir	193.1
Iron	Fe	55.84
Krypton	Kr	82.92
Lanthanum	La	139.0
Lead	Pb	207.20
Lithium	Li	6.94
Lutecium	Lu	175.0
Magnesium	Mg	24.32
Manganese	Mn	54.93
Mercury	Hg	200.6
Molybdenum	Mo	96.0
Neodymium	Nd	144.3

	Symbol	Atomic Weight
Neon	Ne	20.2
Nickel	Ni	58.68
Niton (radium emanation)	Nt	222.4
Nitrogen	N	14.01
Osmium	Os	190.9
Oxygen	O	16.00
Palladium	Pd	106.7
Phosphorus	P	31.04
Platinum	Pt	195.2
Potassium	K	39.10
Praseodymium	Pr	140.9
Radium	Ra	226.0
Rhodium	Rh	102.9
Rubidium	Rb	85.45
Ruthenium	Ru	101.7
Samarium	Sa	150.4
Scandium	Sc	44.1
Selenium	Se	79.2
Silicon	Si	28.3
Silver	Ag	107.88
Sodium	Na	23.00
Strontium	Sr	87.63
Sulfur	S	32.06
Tantalum	Ta	181.5
Tellurium	Te	127.5
Terbium	Tb	159.2
Thallium	Tl	204.0
Thorium	Th	232.4
Thulium	Tm	168.5
Tin	Sn	118.7
Titanium	Ti	48.1
Tungsten	W	184.0
Uranium	U	238.2
Vanadium	V	51.0
Xenon	Xe	130.2
Ytterbium (Neoytterbium)	Yb	173.5
Yttrium	Yt	88.7
Zinc	Zn	65.37
Zirconium	Zr	90.6

INDEX

Page

Aging Oven, photograph of ... 40
Tests ... 43
Alkali Titration Method for the determination of Phosphorus ... 161
Allen Method, determination of Nitrogen in Iron and Steel ... 151
Alternating Stress Tests—Landgraf-Turner ... 69
Testing Machine, photograph of ... 68
Aluminum in Iron and Steel, determination of:
By Bureau of Standards Method ... 169
By Kichline Method ... 75
Armco Ingot Iron, micrograph and analysis of ... 138
Ancient Irons and Modern Research ... 11
Arsenic in Iron and Steel, determination of by Distillation Method ... 77
Atomic Weights, Table of ... 214

Bessemer Steel, micrograph and typical analysis of ... 144
Billhook of Ancient Origin, photograph and micrograph of ... 24
Bismuthate Method for the determination of Manganese ... 139
For the determination of Chromium ... 122
Boron in Iron and Steel, determination of ... 79
Brinell Hardness Test ... 67
Testing Machine, photograph of ... 62
Brunck's Method for the determination of Nickel ... 147
Bureau of Standards Method for Aluminum in Iron and Steel ... 169
Chromium in Iron and Steel ... 121
Melting Points of the Chemical Elements ... 210
Molybdenum in Iron and Steel ... 145
Silicon in Steel ... 169
Sulphur in Iron and Steel (Gravimetric Method) ... 193
Titanium in Steel ... 169
Vanadium in Iron and Steel ... 121
Zirconium in Iron and Steel ... 169
Burrows' Permeability Apparatus, photograph of ... 42

Cain and Maxwell Method for the determination of:
Carbon in Iron and Steel ... 101
Cain—Electrolytic Hydrogen Generator and Reservoir ... 157
Calibrating Thermocouple, photograph of ... 52
Cannon, Iron Band from—analysis of ... 14
Carbon in Iron and Steel, determination of:
By Cain and Maxwell ... 101
By Colorimetric Method ... 81
By Combustion Method ... 83
By Liquid Air Method ... 85
Carbon Monoxide in Iron and Steel, determination of ... 153

Page

Chemical Analysis ... 73
Elements, melting points of ... 210
Chisel of Ancient Origin—Photograph and Micrograph of ... 20
Chrome-Vanadium Steel, determination of phosphorus by Hagmaier Method ... 163
Chromium and Vanadium, determination of, by Bureau of Standards Method 121
Chromium in Iron and Steel, determination of:
By Bismuthate Method ... 122
By Demorest Method ... 123
Conductivity and Permeability Tests, photograph of ... 44
Copper in Iron and Steel, determination of:
Bureau of Standards Method ... 145
Colorimetric Method ... 125
Iodide Method ... 127
Core Loss Test ... 37
Corrosion, Research on ... 15
Cushman Method for the determination of Spelter Coating ... 179

Data—Useful ... 207
Delhi, India, Iron Pillar of—Photograph of ... 8
Delhi, India, Iron Pillar—Analysis of ... 9
Demorest's Method, determination of Chromium and Vanadium in Iron and Steel ... 123
Dougherty's Method, determination of Vanadium in Iron and Steel ... 205
Ductility Tests—Erichsen Method ... 69

Electric furnace, Experimental, photograph of ... 70
Electrical Steels—Magnetic Testing of ... 37
Electrochemical Series, Table of ... 213
Electrolytic Resistance Method for determining Carbon ... 101
Elements, Chemical, melting points of ... 210
Periodic Classification of ... 211
Epstein Testing Coils ... 38
Erichsen Method, for Ductility Tests ... 69
Etching Methods ... 59
Evolution Method for the determination of Sulphur in Iron and Steel ... 191
Experimental Furnace Room ... 71
Heat Treatment ... 71

Fairbank's House, Iron Nails from—Analysis of ... 18
Furnaces, Electric, Experimental, photograph of ... 70

Gauge, U. S. Standard for sheet, plate iron and steel ... 212
Gravimetric Method (Brunck's) for the determination of Nickel in Iron and Steel ... 147
Gravimetric Method for the determination of Sulphur in Iron and Steel ... 193
Gravimetric Method for the determination of Iron in Iron and Steel ... 133

Hagmaier Method for the determination of phosphorus in Chrome-Vanadium Steel ... 163
Hardness Tests ... 67
Heat Treatment, Experimental ... 71
Scientific ... 51
Hydrochloric Acid Method for the determination of Spelter Coating ... 183

Page

Hydrogen in Iron and Steel, determination of ... 129
Hydrogen Electrolytic Generator and Reservoir—J. R. Cain's ... 157

Ingots, Split ... 16
International Atomic Weights ... 214
Introduction ... 5
Iron, determination of in Iron by Gravimetric Method ... 133
Iron Nails taken from Grave ... 28
Iron—Specimens of Old Iron:
Band from British Cannon ... 14
Billhook ... 24
Chisel ... 20
From Merrimac Gunboat ... 26
Nails, of Ancient Origin ... 22
Nails, hand forged used in Mission ... 12
Nails, from Fairbank's House ... 18
Nails, from Bakersfield Weir ... 34
Newburyport Link ... 142
Pillar of Delhi, India ... 8
Yarning Tool ... 84

Kichline Method, determination of Aluminum in Iron and Steel ... 75

Landgraf-Turner—Alternating Stress Tests, photograph of ... 68
Alternating Stress Tests ... 69
Lead-Coated Sheets, sampling and analysis of:
Weight of Coating ... 197
Pin Hole Test ... 165

Magnetic Testing ... 37
Aging Tests ... 43
Core Loss Tests ... 37
Permeability Tests ... 43
Manganese in Iron and Steel, determination of:
By Bismuthate Method ... 139
By Color ... 141
By Persulphate Method ... 137
Test to determine whether Metal is Iron or Steel ... 143
Maxwell and Cain, Bureau of Standards Method for the determination of Carbon in Iron and Steel ... 101
Melting Points of the Chemical Elements ... 210
Merrimac Gunboat, iron from ... 26
Metallurgical Control ... 47
Micrographs:
American Ingot Iron ... 138
Bessemer Steel ... 144
Billhook ... 24
Chisel ... 20
Nails ... 22
Newburyport Link ... 142
Showing the effect of Annealing on hard drawn Wire ... 64
Of Steel—Properly and Improperly Annealed ... 54
Microscopic Tests ... 57

Page
Microscope, view of Apparatus 56
Molybdenum in Steel, determination of, Bureau of Standards Method 145

Nails, analysis of:
Iron Nails of Ancient Origin 22
From Bakersfield Weir 34
From Fairbank's House 18
Hand Forged Nail used in Mission 12
Newburyport Link:
Analysis of 142
Micrograph of 142
Nickel in Iron and Steel, determination of:
Brunck's Gravimetric Method 147
Titration Method 149
Nitrogen in Iron and Steel, determination of 151

Optical Pyrometers 55
Oxidation Method for the determination of Sulphur in Iron and Steel 195
Oxygen in Iron and Steel, determination of 153

Permeability Apparatus, Burrows—photograph of 42
Permeability and Conductivity Tests—photograph of 44
Permeability Tests—Electrical Steels 43
Periodic Classification of the Elements 211
Persulphate Method for the determination of Manganese in Iron and Steel 137
Phosphorus in Iron and Steel, determination of:
Alkali Titration Method 161
Determination of in Chrome-Vanadium Steel 163
Physical Tests 67
Physical Testing Section, photograph of 60
Pin Hole Test—Lead Coated—Tin and Terne Plate 165
Polishing Equipment, photograph of 58
Polishing Methods 59
Pyrometry—Optical and Thermoelectric 55

Recording Potentiometer, photograph of 50
Research on Corrosion 15

Scientific Heat Treatment 51
Section through Split Ingots 16
Sheet Gauge—U. S. Standard for Sheets, Plate Iron and Steel 212
Silicon in Iron and Steel, determination of 167
Silicon in Steel, determination of, Bureau of Standards 169
Spelter Coating, determination of on sheets and wire 183
Cushman Method 179
Lead Acetate Method 189
Spikes, Failure of Steel, photograph and analysis of 32
Steel Pipe Failure, photograph and analysis of 30
Sulphur in Iron and Steel, determination of 191
Bureau of Standards Method 193
Evolution Method 191
Gravimetric Method 193
Oxidation Method 195

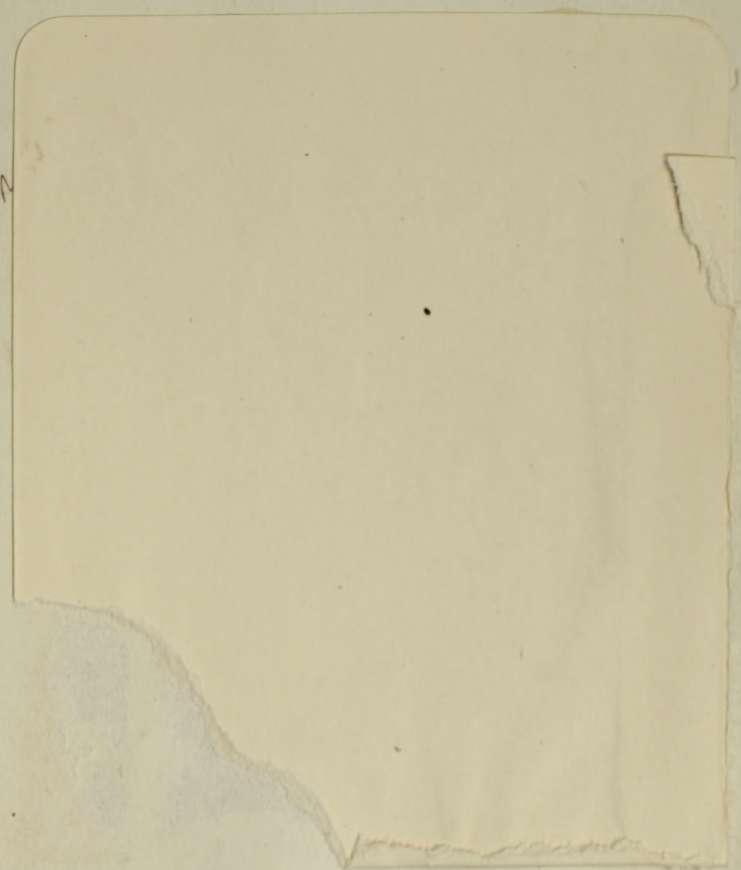
Page
Table of Electrochemical Series 213
Atomic Weights 214
Conversion of Fahrenheit and Centigrade Temperatures 208
Melting Points of the Chemical Elements 210
U. S. Standard Gauge for Sheet Metal 212
Tensile Tests 67
Terne Plate—Analysis of—Weight of Coating 197
Pin Hole Test 165
Testing Coils, Epstein Core Loss Tests, photograph of 38
Thermocouples, Calibrating—photograph of 52
Thermoelectric Pyrometers 55
Tin Plate—Analysis of—Weight of Coating 197
Pin Hole Test 165
Titanium in Steel, determination of:
Bureau of Standards Method 169–203
Titration Method for the determination of Nickel 149

Useful Data 207

Vanadium in Iron and Steel, determination of:
Bureau of Standards Method 121
Demorest's Method 123
Dougherty's Method 205

Weights—Atomic, Table of 214
Wire, Hard Drawn and Annealed, micrographs of 64

Yarning Tool—photograph and analysis of 84
Yensen, T. D., determination of Carbon in Iron 85

Zirconium—determination of—Bureau of Standards Method 169

in-docu